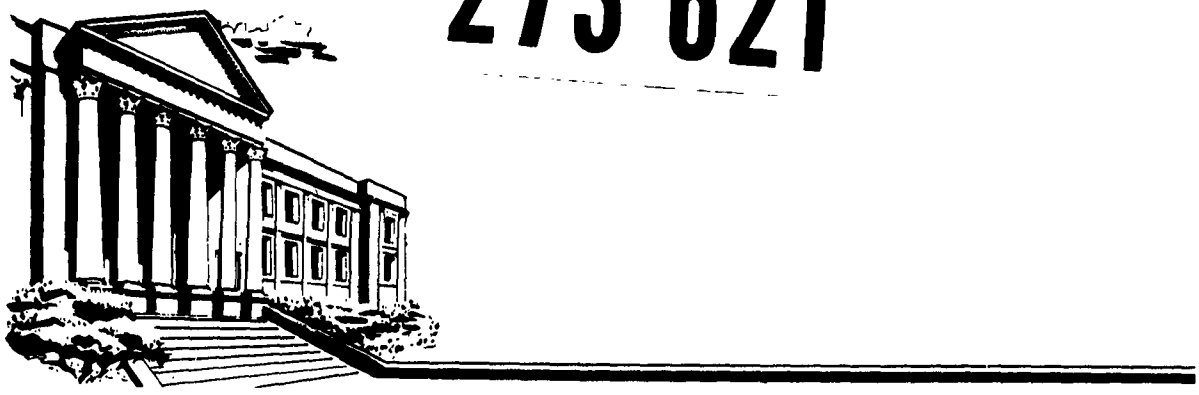

Final Report F-A2476

PREPARATION AND EVALUATION OF HIGH PURITY BERYLLIUM

by

Grant E. Spangler
Marvin W. Herman
Edward J. Arndt

November 2, 1960 to November 1, 1961

Prepared for

DEPARTMENT OF THE NAVY
Bureau of Naval Weapons

Contract No. NOw 61-0221-d

THE FRANKLIN INSTITUTE
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ABSTRACT

Zone refining procedures for vertical floating-zone type zone melting of beryllium are described. Single crystals prepared from zone melted beryllium were tested in tension with their orientation arranged to yield basal plane slip. The critical resolved shear stress varied from 2400 to 400 PSI, decreasing with increased purification; the glide strain varied from 16 to 220% increasing with increased purification. Procedures for the rolling of several single crystals of beryllium are described and the results of recrystallization studies on them are presented.

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I. INTRODUCTION

The purpose of this research is to study the deformation and fracture characteristics of beryllium. The desired goal is a mechanistic explanation for the brittleness or lack of ductility usually observed in the metal. This report represents the second year's effort of this investigation and deals with the preparation of high purity beryllium by zone melting and the mechanical property evaluation of this material in single crystal and to a limited extent in polycrystalline form.

The room temperature deformation characteristics of polycrystalline beryllium as observed by many workers have clearly shown that the ductility of the material strongly depends upon both grain size and the degree of preferred orientation. With suitable grain size and the orientation of the grains arranged to restrict slip on the basal planes and permit slip on the prism planes, appreciable ductility is observed. For example, in hot extruded and cross rolled sheet most of the crystals are oriented with their basal planes lying in the plane of the sheet and as much as 40% elongation can be attained in the longitudinal direction. The available extension in the transverse directions is considerable less and is essentially non-existent in a direction normal to the rolling plane.

By examining the work on the deformation modes of single crystals[1,2,3,4] at room temperature one sees that this strong plastic anisotropy is to be expected. In particular, the limited ductility obtained by Tuer and Kaufmann[2] in single crystals oriented for basal slip appears to explain much of the brittleness observed in unfavorably oriented polycrystalline material. This limited slip prior to fracture, at least with crystal orientations comparable to Tuer and Kaufmann, was also observed and recently reported by Garber, Gindin, and Shubin[4]. The beryllium used by them was purified by vacuum distillation and appeared to be appreciably purer than the beryllium used by Tuer and Kaufmann. It would seem that the brittleness associated with basal glide

was not affected by purification. Our previous studies[5,6] on zone refined single crystals of beryllium have indicated, however, that a high degree of basal plane ductility is possible and that this probably is related to purity. The degree to which this ductility could be extended, along with a clear delineation of the factors responsible for it and its resultant effect on polycrystalline properties were the aims of this current investigation.

In this report the methods and results of the work to refine beryllium and examine its properties will be presented. The refining of the beryllium by zone melting includes a description of the equipment and the procedures used and a discussion of the manner in which impurity redistribution and removal occurred. The evaluation of the mechanical properties of the zone refined beryllium includes a description of the equipment and techniques used to prepare and test single crystal samples, the results of the tests, and a discussion of proposed effects of impurities and testing conditions on a fracture criterion applicable to beryllium. Also included are the procedures used to prepare polycrystalline beryllium from single crystals and the results of several bend tests of this material.

2. ZONE REFINING

2.1 Procedures

During the past year a series of one quarter inch diameter and one inch diameter beryllium bars have been zone refined by the vertical floating zone technique. Listed below are several different starting materials of varying degrees of purity which have been used.

Quarter Inch Bar Starting Stock

a) HP Grade Pechiney flake, vacuum cast and extruded
b) SR Grade Pechiney flake hot extruded
c) NMI vacuum distilled material, vacuum cast and extruded

One Inch Bar Starting Stock

a) Brush pebble,vacuum cast
b) HP Pechiney powder, hot extruded.

These bars have been subjected to various numbers of zone passes and have been evaluated at various stages of refining, principally with respect to their mechanical properties. A complete summary of these bars and their histories appears further on in this report.

Briefly summarizing, (see App. I for details) the zone melting was carried out in all glass hermetically sealed systems containing a gettered argon atmosphere at slightly less than one atmosphere pressure. One half an inch per hour travel rates were employed, with the exception of the initial passes through several bars where excessive volatilization required speeds at 1 1/2"/hour for successful viewing of the zone. Seeding of single crystals was carried out, when necessary, to produce desired orientations.

The all glass system used for the one quarter inch bars is shown in Figure 1. The development and operation of this system has been described previously[5]. The apparatus for the one inch diameter bars was developed during the present contract year and is described in detail in Appendix I.

2.2 Results and Discussion

2.2.1 Redistribution of Impurities by Zone Melting

Although the analytical results on the zone refined beryllium are somewhat limited there is little doubt that redistribution of many of the impurities has occurred. Both mass spectrograph analyses and emission spectrograph analyses have been made on the zone refined beryllium and demonstrate this effect (see Table I and II). An examination of the mass spectrograph analyses shows that the principal impurities, Fe, Si, Al, Mn, and C all tend to segregate to the finishing end of the zone refined bars, indicating that these elements have segregation coefficients

Table I

MASS SPECTROGRAPH ANALYSIS OF BERYLLIUM[8]

Impurity	Approximate Detection Limit (ppm Atomic)	Line Used for Estimate (Mass Units)	Estimated Impurity Concentrations Berylco Zone-Refined 5 passes: First End to Freeze (ppm Atomic)	Last End to Freeze (ppm Atomic)	Pechiney Powder Rod (ppm Atomic)
Carbon	1	12	180	400	600
Nitrogen	1	14	< 10	< 10	< 25
Oxygen	1	16	250	440	600
Fluorine	0.03	19	1	1	4
Sodium	0.03	23	1	1	8
Magnesium	0.03	24, 25, 26	< 0.3	< 0.3	20
Aluminium	0.03	27	1	35	600
Silicon	0.03	28 (29)	< 10	< 20	~ 80
Phosphorus	0.02	31	0.2	< 0.2	2
Sulphur	0.02	32	2	3	6
Chlorine	0.02	35	1	< 0.2	4
Argon	0.01	40	< 6	< 7	< 30
Potassium	0.01	39	1	0.6	1
Calcium	0.01	40	< 6	< 7	< 30
Titanium	0.01	48	< 0.1	0.2	3
Vanadium	0.01	51	< 0.1	0.2	0.1
Chromium	0.01	52	< 0.1	0.2	4
Manganese	0.01	55	0.2	4	10
Iron	0.01	(54) 56 (57)	40	100	63
Cobalt	0.01	59	0.2	0.2	0.2
Nickel	0.02	58, 60 (62)	46	21	17
Copper	0.02	63, 65	12	12	1
Zinc	0.02	64, 66, 68	0.5	< 0.2	< 0.2
BeH	0.1	10	50	150	30
BeO	0.03	25	< 8	< 15	< 4
Be_2O	0.02	34	4	6	4

Table II

EMISSION SPECTOGRAPHIC ANALYSES OF BERYLLIUM*
(weight parts per million)

Element (ppm)	8 Zone Pass HP Pechiney Rod		One Zone Pass SR Pechiney Rod	Starting SR Pechiney Rod
	Starting Region	Finishing Region		
BeO				3100
C				90
Al	< 40	40	40	40
Cr	inv.	inv.	tr	tr
Fe	<< 40	150	40	200
Mg	6	4	4	4
Mn	<< 10	<< 10	15	15
Ni	40	20	40	40
Ca	< 30	< 30	< 30	< 30
Cu	4	4	8	8
Si	60	< 60	< 60	< 60
Na	<< 30	<< 30	<< 30	<< 30
Zn	< 40	< 40	< 40	< 40
Pb	inv.	inv.	inv.	inv.

inv. = undetected
tr = traces
<< = Very much lower

* These analyses were performed through the courtesy of the Pechiney Laboratory at Chambery

less than unity. Nickel tends to segregate to the starting end; this is in agreement with the recently determined Be-Ni phase diagram[7] which shows nickel to have a segregation coefficient greater than unity. Copper is apparently difficult to remove by zone melting in that its segregation is negligible. While magnesium shows no tendency to segregate to either end, it has been reduced to a low level quite effectively through volatilization.

The mass spectrograph has the advantage of utilizing small samples to determine the concentrations of the whole spectrum of impurities found in beryllium. It also shows promise of being an excellent means of attacking the difficult problem of analyzing for oxygen in beryllium. Some effort must be spent, however, in correlating the mass spectrograph analyses with other conventional techniques in order to determine with certainty that all impurity isotopes are created in proportions representative of their concentrations in beryllium.

The mass spectrograph analyses were performed on previously zone refined Berylco vacuum cast rod; the emission spectrograph analyses (Table II) were performed on more recently zone refined Pechiney HP and SR grade beryllium. These latter materials have lower initial impurity concentrations than the Berylco material and result in the low impurity levels attained after zone melting.

It is of importance to note in these analyses the effectiveness of the removal of iron, especially in the sharp drop exhibited by the SR grade material after only a single pass. This is particularly significant when the results of one zone pass on distilled beryllium is considered.

A portion of a sample of distilled beryllium* which was vacuum melted and extruded was zone melted with one pass at 1 1/2" per hour at this laboratory to form a 45° orientation single crystal. The crystal

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* This beryllium was produced at N. M. I.

was pulled to fracture and the fracture surface examined. The number of precipitates on the fracture surface of the type which Levine has described (see section 3.2.3) and feels adversely affects ductility was significantly reduced over the number that appeared on the fracture surface of the material prior to zone refining. Iron, according to Levine is one of the constituents of the fine precipitates as determined by micro-beam analysis. Thus, one zone pass even at a relatively rapid rate is effective in reducing the iron content and amount of precipitates felt to lead to brittle behavior.

In addition to the normal purification achieved by zone refining, demonstrated by these analyses, it is also important to note several additional methods of purification afforded by zone melting. These are the ability to purify by the agglomeration of insoluble phases at the surface of the molten zone where they can subsequently be removed, and by the transporting of insoluble phases along with the rapidly stirred molten zone. These effects, along with evidence of purification by volatilization are described in the following section by detailed observations of the first few passes through one of the one inch diameter vacuum cast Brush bars.

2.2.2 Redistribution of Impurities by Agglomeration and Volatilization

The total length of the bar was twelve inches, with one inch at each end of the bar held in titanium chucks. The first pass was made from a point three and one half inches from the top chuck and traversed four and one half inches along the bar to a point two inches from the bottom chuck. During this first pass a very heavy oxide or drossy layer collected on the surface of the molten zone. In addition, a rather high rate of vapordeposition occurred on the inner surface of the quartz enclosure tube making it necessary to utilize the relatively rapid rate of zone travel of one and one half inches per hour to maintain visibility of the zone. After completion of the first pass the heavy dross collected on the surface of the bar was removed by pickling in a nitric, hydrofluoric acid bath, leaving a pitted surface on the bar.

A second pass was then made starting two inches from the top chuck and traversed six inches to the previous end position, two inches from the bottom chuck. The initial one and one half inches of this pass had not previously been melted and was identical in character to the first pass; it exhibited heavy dross on the surface and high rate of vapor deposition. Upon reaching the portion of the bar previously melted there was a very sharp reduction in both the amount of dross on the surface of the zone and in the rate of vaporization from the melt. After completion of this pass the bar was again heavily etched. As seen in Figure 2, the two regions of the bar are quite distinct; the heavily pitted singly-melted section is at the left and the doubly-melted section where only occasional pitted bands appear is at the right. The bar in Fig. 2 is obliquely lighted to accentuate the pitted regions and t[illegible]ight, shiny areas appear dark.

A third pass was then made at one half inch per hour on the bottom four and one half inch section. During this pass the molten zone was largely free of dross during most of the pass. The majority of the dross that did form was carried along on the surface of the molten zone and deposited at the last zone to freeze. In Figure 3 the bar with three passes is shown after etching. The single pass, heavily pitted region is to the left. The surface of the three pass section is now smooth, bright and almost mirror like. The only pitting in this region is observed at the final zone to freeze. The bar has long columnar grains throughout its length (not visible with this etchant) and also exhibits hot cracking at grain boundaries in the finishing region of the bar; one of the cracks may be seen at the right hand end of the melted bar.

In Figure 4 the bar is shown etched after a fourth pass has been made through the final four and one half inches of the bar. This section of the bar is now a single crystal. The facets visible on the surface of the bar are related to crystallographic planes of the crystal.

In Figure 5 the grain structure of the same bar is revealed by a NH_4Cl-HCl etch. At the far left is the original equiaxed cast structure, next the long columnar grain structure of the single pass region and then the single crystal four pass region.

These four passes demonstrate quite markedly the ability of vertical zone melting to purify by (1) agglomeration of insoluble phases at the surface where their removal is possible by etching, (2) volatilization of high vapor pressure impurities as evidenced by a sharp drop in the rate of vapor deposition after the initial pass, and (3) the transporting of insoluble phases along with the rapidly stirred molten zone as evidenced by the dross collected at the final zone to freeze.

It should be pointed out, however, that there is an apparent limit to the amount of insoluble phases, in particular BeO, that can be removed by agglomeration or transport. In Figure 6 is shown a photograph of a 1/4 inch diameter commercial purity hot pressed and extruded bar which has been used as a gettering bar. This bar originally contained the normal 2% BeO content. As zones were passed through this bar (from left to right on photograph) there was initially some tendency for the oxide to agglomerate at the surface and to travel with the zone. A point was reached, however, along the bar where the oxide accumulation extended through the volume of the molten zone and produced an extremely mushy condition in the molten zone. When this occurred the oxide could no longer move to the surface nor travel with the zone and a more or less static condition prevailed. As the zone moved further along it again reached an "unsaturated" region and the cycle repeated itself with the oxide moving until the mushy build-up occurred again. This behavior is visible in the photograph as the intermittent bright, shiny areas and dull regions along the length of the bar. The dull regions are the "saturated", high oxide sections. Although this particular bar has been subjected to more than five passes, the mushy regions continue to persist during melting, demonstrating the static nature of these areas.

It appears that this accumulation type of behavior occurs in materials containing greater than 0.5% BeO. A good example of this was found in an attempt to zone melt a one inch diameter hot pressed and extruded Pechiney powder rod. This particular bar contained 0.7% BeO, which is considerably less than that found in the standard commercial powder rod, and is just slightly higher in BeO content than that of successfully zone melted vacuum cast material. After three passes the bar, shown in Figure 7, still exhibited a heavily drossed surface and mushy regions similar to those observed in the commercial powder rod. This condition made it necessary to discontinue zone melting of this particular bar.

Usually, after about five passes a condition is reached where the bar is bright and mirror-like after etching. Upon remelting, however, small amounts of insoluble patches, probably oxides, reform on the surface of the melt. While there is some tendency to carry these along in the zone they are largely left behind on the surface of the solid bar. These patches which can readily be removed by etching but reform on melting can apparently be attributed to the formation of surface oxide or hydroxide upon exposure of the freshly etched surface to the wash water or to air. It has not been possible to devise a technique for removing the patches without creating a condition such that new ones form on melting.

This observation infers that the molten zone is always saturated with oxygen. What the solubility of BeO in Be is, no one knows or will know until improved analytical techniques are developed. The present analytical limit for BeO is on the order of several hundred ppm and does not distinguish between BeO and dissolved oxygen. This lack of analytical facility not only makes it difficult to determine what is happening to the BeO content during zone melting but also, by preventing the determination of phasal relationships between Be and BeO, makes it impossible to predict what should be expected with regards to BeO segregation during zone melting.

3. MECHANICAL PROPERTY EVALUATION OF SINGLE CRYSTALS

3.1 Tensile Testing Procedures and Results

In order to evaluate the mechanical properties of the zone refined beryllium, a series of single crystal tensile tests have been performed. The orientation of the crystals has been chosen to result only in basal slip. The tensile specimens were, in most cases, prepared by a combination of spark discharge machining and electro-chemical machining. The spark discharge machining apparatus and the machining procedures are described in detail in Appendix II. The tensile specimens had gage sections one-half inch long and approximately 0.100 inches in diameter, with conical shaped shoulders at the extremities for gripping, see Figure 8. The specimens were tested in an Instron tensile machine at a strain rate of 7×10^{-4}/sec., using two short lengths of steel chains at each end of the tensile grips in order to minimize non-axiality during loading, see Fig. 9.

The results of these tests are summarized in Table III along with the preceeding zone melting history of each specimen. In Figure 10 are shown a series of typical shear stress - shear strain tensile curves.

3.2 Discussion of Testing Results

The principal feature of the tensile deformation of beryllium by basal glide that has been discovered is clearly revealed in the shear stress-strain diagram of specimen P-1-3 (see Fig. 10). The appearance of an initial work hardening region, an easy glide region, and a region of rapidly increasing stress with strain typical of close packed metals demonstrates without any doubt that there is no intrinsic brittleness associated with basal glide in beryllium.

Table III

TENSILE TEST RESULTS

Specimen Designation	Starting Material	Zone Melting: No. of Passes ½"/hr	Zone Melting: Position in Bar	Diameter of Gage Section (in)	Orientation Basal Plane	Critical Resolved Shear Stress (psi)	Overall Elongation (%)	Total Glide Strain (%)	Glide Plane	Fracture: Crystal Plane	Fracture: Position on Tensile Bar
B-2-1	Berylco Vac. Cast	2	Finish end	0.105	$\chi_o = 47°$ $\lambda_o = 51°$	2400	9	16	Basal	Basal	Center
B-1-1	" " "	5	Center	0.140	$\chi_o = 20°$ $\lambda_o = 25°$	1350	24	64	Basal	Prism	Shoulder
P-1-3	Pechiney Vac. Cast	5	Center	0.064	$\chi_o = 48°$ $\lambda_o = 52°$	680	140	220	Basal	Basal	Shoulder
P-3-3	" " "	8	Finish end	0.107	$\chi_o = 46°$ $\lambda_o = 53°$	900	10	22	Basal	Basal	Center
P-3-1	" " "	8	Starting end	0.102	$\chi_o = 48°$ $\lambda_o = 55°$	520	82	140	Basal	Basal	Center
P-3-2	" " "	8	Center	0.102	$\chi_o = 45°$ $\lambda_o = 50°$	690	(c)	(c)	Basal	(a)	(c)
P-1-1	" " "	5	Finish	0.110	$\chi_o = 47°$ $\lambda_o = 51°$	600	(c)	(c)	Basal	(c)	(c)
Gettering Bar	" " "	———	———	0.082	$\chi_o = 12°$ $\lambda_o = 29°$	1900	(c)	(c)	a. Basal	Prism	Center
P-1-2	" " "	5	Starting end	0.114	$\chi_o = 7°$ $\lambda_o = 19°$	1690(e)	(c)	(c)	b. Prism (e)	(c)	(c)
P-5-1	" " "	1(d)	Starting end	0.102	$\chi_o = 47°$ $\lambda_o = 52°$	1090	82	140	Basal	Basal	Center
V-C				0.105	$\chi_o = 45°$ $\lambda_o = 45°$	2600	2	4	Basal	Basal	Center
4PB	Brush Vac. Cast	4(a)	Starting end	0.116	$\chi_o = 74°$ $\lambda_o = 74°$	1070	(c)	(c)	Basal	(c)	(c)
VD-1-P	NMI Vac. Distilled	1(d)	Starting end	0.1225	$\chi_o = 45°$ $\lambda_o = 50°$	905	32	61	Basal	Basal	Shoulder
VD-7-P	" " "	7	Starting end	0.080	$\chi_o = 50°$ $\lambda_o = 56°$	400	(c)	(c)	Basal	(c)	(c)
SR-1	Pechiney SR Grade	3(b)	Starting end	0.116	$\chi_o = 48°$ $\lambda_o = 52°$	803	(c)	(c)	Basal	(c)	(c)

(a) Two Passes - 1½"/hr
One Pass - ½"/hr - 2 hours
One Pass - ½"/hr

(b) Two Passes - 2"/hr
One Pass - ½"/hr

(c) Not pulled to fracture

(d) Seeding Pass - ½"/hr - 15 to 30 min
1½"/hr - remainder

(e) At this orientation both basal and prism slip are equally likely and slip traces of both were seen soon after yielding; resolved shear stress on the prism plane at yielding was 7670 psi.

As will be discussed in subsequent sections, the brittleness of single crystals observed by Tuer and Kaufmann[2] was produced by impurities, while that observed by Garber, Gindin, and Shubin[4] was probably the result of their method of preparing and testing single crystals. It is evident from Figure 10 that all the specimens tested did not produce the aforementioned deformation sequence, nor did they exhibit the extensive ductility of P-1-3. In the following sections the factors felt to be responsible for the observed differences in behavior will be discussed. In brief they deal with the effects of impurities and the effects produced by the manner in which the tensile tests were conducted on a fracture criterion[9] that appears applicable to beryllium. For purposes of clarity the results are first dealt with in terms of the qualitative aspects of the criterion and then those aspects that can be quantitatively treated are presented. Also a discussion on absolute purity level and ductility and the relationship between single crystal and polycrystalline ductility is included.

3.2.1 The Impurity Effect in Beryllium

The refining action of zone melting in removing many impurities is evident from the results of the chemical analyses (see Table I and Table II) and in the variation of the critical resolved shear stress for yielding (see Table III). In the latter case it is assumed that the level of the yield stress depends upon "strengthening" by impurity atoms; the correlation between the extent of zone refining (number of passes and position along the bar) and the yield stress is then evidence of purification. It is, therefore, suggested that without regard to the way in which impurities are interacting with the deformation there exists a marked adverse impurity effect on the basal plane ductility of beryllium.

3.2.2 Effect of Purity on Ductility and Rate of Work Hardening

The detailed manner in which impurities and testing conditions are expected to affect ductility is examined by considering their effect on a deformation model which can be used to describe crack initiation and propagation in beryllium.

Tuer and Kaufmann[2] observed that fracture by basal plane cleavage was always associated with the prior formation of bend planes. This cracking, designated by them as the "kink, cleavage-plane defect", appears to arise out of the stresses developed at the base of a bend plane according to a model proposed by Orowan[10]. The misorientation of the basal planes across the band plane necessary to develop sufficient stress to nucleate cleavage has been estimated to be 5° by Friedel[11]. Stroh[9] proposes that the termination of a bend plane within a crystal can arise by a bend plane splitting. He examines the condition for the propagation of a crack formed when a bend plane (or wall of dislocations) has split and derives a fracture criterion for metals that slip and cleave on the same plane. This failure criterion has been applied by Stroh to a study of the fracture of zinc at -196°C[12]. It has described the observed effect of orientation on fracture and yields an experimental constant which compares very well to a theoretical one.

Green and Sawkill,[13] pointing out the similarity of mode of fracture of beryllium and zinc, applied the Stroh criterion to beryllium. By assuming values for the surface energy and a hardening parameter they estimated the ductility of beryllium during basal glide and found it was comparable to that observed by Tuer and Kaufmann. Assuming operation of the Stroh fracture model, Green and Sawkill suggested that the relative brittleness of beryllium is the result of a high yield stress and that improvement in the ductility is possible if the yield stress could be reduced by purification. Implicit in their suggesting that a decrease in yield strength could result in an increase in ductility is that no compensatory increase in work hardening occurs.

It is seen in Figure 10 that there is a general increase in ductility with a decrease in both the rate of work hardening (at least in the early stages) and the yield stress. It is felt that this is the result of the purifiying effect of zone melting. The apparent inconsistencies in some of these ductility results can be resolved when the additional ductility influencing effects of the testing variables are considered.

3.2.3 Effect of Testing Variables on Ductility

3.2.3.1 Size Effect

Stroh[9] points out that smaller diameter crystals should sustain greater fracture stresses. This has been presented in terms of strain by Green and Sawkill[13] who noted that an appreciable increase in ductility is to be expected with a decrease in diameter of crystals having similar work hardening characteristics and similar yield stresses. This appears to be the case when specimen P-1-3 and P-3-1 are compared (see Fig. 10 and Table III). Although this apparently explains why the specimen with the highest yield stress was the more ductile, examination (next section) of the sequence and position of fracture of both specimens suggested that still a second factor enhanced the ductility.

3.2.3.2 Effect of Bending Constraints

To present this properly it is necessary to note that the Stroh analysis for fracture requires the prior nucleation of a crack by bend plane splitting. In the extreme case where no bend planes are formed or where no obstacle exists to "split" a wall of dislocations, plastic flow could continue to a shear rather than a cleavage fracture. Considering only bend plane formation, the value of ductility observed in a tensile test of a single crystal of a metal such as beryllium should reflect in part the extent of bending introduced by the test conditions.

Specimen P-3-1 fractured in the center of the gage section (see Fig. 11) which is a region where kink bands (double bend planes) are expected to form due to the reverse bending in this region developed by the constraining effect of the grips*. Further, no cracks which, as explained shortly, could relieve this bending were observed in the shoulder sections in the grips. Specimen P-1-3 in contrast failed in the grips by basal plane cleavage (see Fig. 12). The basal plane cracks which were responsible for the cleavage developed in the shoulders early in the deformation and were associated with electro-spark discharge machined surfaces which had not been removed by electro-polishing. These cracks which continuously opened up during the deformation appeared to act as self-aligning devices by tending to reduce the bending constraint effects of the grips on the section in the gage length where kinks should form.

One can visualize this reduction of the reverse bend stresses in the center of the gage section by considering the bending moment required at the grips to close an already formed crack in the grip section. This is equivalent to having no crack form in the first place. The application of an external bending moment to close the crack is resisted by the gage section, hence the bending in the gage section increases. It is therefore conceivable that the additional bending developed in P-3-1 because no cracks formed in the grips led to the formation of bend planes earlier in the deformation and hence in part explains its lesser ductility. There remains, however, another contributory factor to the apparent premature failure of P-3-1; it is considered next.

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* Bend planes also form at the ends of the gage section next to the grips but are not likely to produce fracture by a splitting operation because, in contrast to the region where kinks form, no shearing across them can develop.

3.2.3.3 Effect of Surface Damage

A third contributory effect which can influence ductility is the initiation of cleavage cracks by surface damage. The relatively limited slip observed in P-3-3 and P-1-3, in view of their low yield stresses, is felt to be caused in part by accidental surface damage. In the region of the crystals close to the final fracture surface, several cleavage cracks were found on P-3-3; one small one was found on P-3-1. Each crack was associated with plastic deformation of the surface layers that had occurred prior to testing. It is not known whether the cracks formed prior to testing or during testing.

3.2.3.4 Effect of Orientation

It is noted that specimen B-1-1 cannot be compared directly to the others because its orientation ($\chi_o = 20°$) should permit it, according to theoretical predictions [9,13], to exhibit a ductility greater than that of a 45° crystal of similar yield strength. (The latter orientation is typical of the other specimens tested).

Summarizing briefly, the general relationship of increased ductility with increased purity (lower yield stress) appears more reasonable when some account is made for the behavior of specimens P-3-3 and P-3-1. Further, this relationship appears in qualitative agreement with predictions derived from Stroh's fracture criterion.

3.2.4 Effect of Precipitates

The effect of precipitates on ductility will be examined by discussing the following pertinent observations:

a) large inclusions can nucleate cleavage cracks.
b) small precipitates (~ 5 microns), hexagonal in shape and apparently easily cleaved, do not appear to limit ductility.
c) small precipitates (~ 1-2 microns), spherical in shape and not easily fractured, do appear to limit ductility.

The test on crystal V-C revealed that large precipitates appear effective in crystals of this purity in initiating cleavage. An examination of the surface of this vacuum cast crystal revealed a basal cleavage crack at an inclusion close to the final fracture surface (see Fig. 13). No similar phenomenon was observed on any of the zone melted crystals. It could not be determined whether the crack at the inclusion arose from a bend plane or dislocation wall splitting phenomenon or from a stress concentration developed by the presence of the inclusion.

An examination of the fracture surface of P-5-1 revealed uniformaly distributed precipitates about 5 microns in size and hexagonal in shape. No identification of them was made. There were about 100 precipitates in the fracture surface (about 6 mm^2). The matrix material adjacent to the precipitates did not appear torn or deformed; the edges of surface of the precipitates appeared in the plane of the fracture surface. It was reasoned that the precipitates cleaved readily when the cleavage crack propagating through the matrix reached them. There appeared very few precipitates of the type described in the following paragraphs. Since P-5-1 exhibited 82% overall elongation, it is felt these precipitates did not limit ductility.

A type of precipitate felt to limit ductility has been described by Levine[14]. In the evaluation of polycrystalline beryllium produced by vacuum distillation, Levine pointed out that a bend specimen showing approximately 20% elongation in the outer fibers at fracture was characterized by a minimal amount of very fine spherical precipitates (1-2 microns) on the fracture surfaces. The matrix material adjacent to the precipitates was torn and deformed and the precipitates were not in the plane of the surface. Other specimens showing very limited ductility were characterized by large amounts of these fine precipitates on the fracture surfaces. It was concluded that these precipitates strongly influence the ductile behavior of the polycrystalline beryllium.

These type of precipitates appear in crystals P-3-3, B-2-1, and V-C and only to a very limited extent if at all in the other crystals tested. It is reasonable to assume that they appeared in V-C and B-2-1 because these crystals were relatively impure (note high yield points, see Table III). P-3-3 although exhibiting a low yield point was the "finish" end of a zone melted bar and region of the crystal where precipitates could be concentrated.

While the contribution of these precipitates in limiting ductility cannot be estimated, it is felt that in part they did adversely affect the ductility observed in these crystals. The way in which this could occur, although speculative, is briefly mentioned:

1) the precipitates formed in substructure arrays[15] which:
   a) provide an existing tilt boundary for splitting
   b) provide a barrier[9] to split a wall of moving dislocations.
2) the precipitates in a non-uniform distribution provide "hard" and "soft" regions conducive to kinking[16] (double bend planes), the first step in the fracture sequence.

It is clear that some caution must be exercised when general statements regarding the effects of precipitates in beryllium are made.

3.2.5 Fracture Criterion Analysis

In the previous sections the qualitative aspects of the Stroh analysis were used to describe the observed tensile deformation results. In this section the quantitative results of the analysis will be presented.

The equation derived by Stroh to establish a criterion for fracture for metals that flow and cleave on the same plane is presented in the form used by Green and Sawkill[13] in their application of it to beryllium.

$$\frac{(\tau_s - \tau_o)\sigma_n D}{\cos \lambda} = \frac{\gamma}{\pi B} \ln \frac{\theta}{B \tau_s}$$

τ_s = resolved shear stress on the basal plane at fracture

τ_o = critical resolved shear stress on the basal plane (yield stress)

σ_n = resolved normal stress on the basal plane at fracture

D = minor diameter of the crystal = $\frac{Do \text{ (initial diameter)}}{1 + \delta\text{(overall elongation)}}$

λ = angle between the slip direction and the tension axis

θ = angle of misorientation across bend plane (or tilt boundary) that is split

$\frac{2}{B}$ = "a complex function of the elastic constants, roughly equal to a mean elastic extensional modulus."* The value used: 3×10^{12} dynes/cm^2.

Y = energy required to form new surface**. The value used: 1000 ergs/cm^2.

When the value on the left side of the equation is equal to or greater than the right side, the continuous propagation of a crack formed during deformation at a bend plane or tilt boundary of angle θ ensues. Since variations in θ or τ_s in the log term have little effect upon its value, the value on the right hand side is essentially a constant of the material, if Y, the surface energy required to form a new surface during cleavage, is assumed to be independent of purity. The equation cannot be used to predict explicitly a fracture stress unless the shear stress-shear strain curve for the material is known. Thus, to determine if the fracture criterion is applicable, one performs tensile tests as a function of crystal orientation and compares the experimentally determined left hand side of the equation to the "theoretical" constant on the right side. It is preferable to use crystals having the same yield stress and varying orientation, since to vary yield stress it is necessary to vary impurity levels and the explicit effect of impurities

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* Green and Sawkill[13]

** Green and Sawkill[13] suggest that the value for Be and Zn should be in the same ratio, 8.4, as their extensional moduli in the c direction. Y for zinc was obtained from the work of Gilman[17].

on the sequence of bend plane (or tilt boundary) formation and splitting is not known. Unfortunately in the present investigation there is no choice but to use crystals of varying yield stress.

Using a value of θ of approximately 6°* and the extremes of the observed fracture stresses, the value of the theoretical constant ranges from 3.0×10^{15} to 3.5×10^{15} dynes^2/cm^3. The experimental value of the constant for the specimens tested is given in order of decreasing yield stress.

V-C	2.6×10^{15} dynes^2/cm^3
B-2-1	5.3×10^{15} dynes^2/cm^3
B-1-1	3.3×10^{15} dynes^2/cm^3
P-5-1	5.9×10^{15} dynes^2/cm^3
P-3-3	2.2×10^{15} dynes^2/cm^3
P-1-3	1.9×10^{15} dynes^2/cm^3
P-3-1	$.53 \times 10^{15}$ dynes^2/cm^3

In view of all the contingencies of the effects of impurities and testing variable on the sequence of crack initiation and propagation the agreement is remarkable.

Assuming that the Stroh criterion reasonably describes the observed fracture of beryllium, the numbers derived from the analysis lend further support to the previous contention, based on experimental observation, regarding the premature failure of specimens P-3-3, V-C, and P-3-1.

The limited data obtained to date, of course, does not prove that the simple fracture model upon which the Stroh criterion is based is completely applicable. Because of the necessary sequence of events required to cause a fracture, it would be more appealing if there could

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* Friedel[11] estimates this at 5°

be incorporated in the criterion the condition for crack nucleation and the extent of bending uniquely associated with the geometry of the specimens and the grips. In any case, however, the numbers derived from the equation relating the experimental values of the tensile tests appear to indicate that some crystals did fail prematurely and why there is an increase in ductility with purity. By examining the details of the shapes of the stress-strain curves along with the inter-relation of the variables in the fracture criterion equation, it is possible to determine what aspects of the effects of impurities in beryllium are particularly important with regard to ductility.

The first consideration is that of the effect of impurities on yield strength. Assuming only that yield strength varies and that work-hardening is constant, the value of

$$\frac{(\tau_s - \tau_o)\sigma_n D}{\cos \lambda}$$

the experimental fracture relation, depends only upon σ_n. Rewriting the expression noting that $\sigma_n = \tau_s \tan \lambda^*$, yields

$$\frac{(\tau_s - \tau_o)\tau_s \tan \lambda \, D}{\cos \lambda}$$

Thus, the ratio of the value of the relation for two hypothetical crystals of different τ_o but similar work hardening goes as the ratio of their flow stresses at any value of strain. The crystal having the higher initial yield strength is expected to fracture sooner. That is, when the higher yield strength crystal reaches the value of the relation at which fracture is expected to occur, the lower yield stress crystal has a lesser value.

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* This is applicable if $\chi \cong \lambda$

To a first approximation this is seen in the comparison of P-3-3 and B-2-1. These crystals essentially have the same initial orientation, work-hardening character, and ductility. The value of the experimental fracture relation roughly differs in the ratio of the fracture stresses.

Second, is the effect of impurities on the work hardening properties. It is obvious that if linear hardening were observed, with the extent of the hardening increasing with impurity content, the value of the experimental fracture relation at a given strain would increase with impurities not only because of the increase in σ_n with τ_o, but also because of the increase in the value $(\tau_s - \tau_o)$. Actually the effect of decreased work-hardening on increased ductility is much more influential than revealed by this simple consideration of linear hardening. For example, examine curves P-1-3 and P-5-1 in Figure 10. Although both crystals have essentially the same τ_s, shear stress at fracture, P-1-3 not only exhibits a greater ductility, but also has a significantly lower experimental fracture relation value. This result was obtained because of the easy glide region developed by P-1-3. During easy glide appreciable strain occurred at little increase in flow stress. However, the significant point is that the basal plane rotation accompanying the strain reduces both the normal stress component and the maximum length of the dislocation wall that is to be split; i.e., the term $D/\cos \lambda$. The rapid work hardening experienced by P-1-3 in the latter stages of strain is not particularly effective in increasing the value of the experimental fracture relation since the increase in $(\tau_s - \tau_o)$ is offset by the decreasing values of σ_n and $D/\cos \lambda$. Thus, high final fracture strengths may be obtained by virtue of an initial easy glide region.

It is seen, therefore, that the influence of impurities on ductility in beryllium arises out of their effect on the yield strength and work hardening characteristics. The yield strength increase arises out of solid solution hardening (and possibly precipitation hardening);

the increase in work-hardening with impurities is generally observed in alloy systems, but the exact mechanism is yet unknown. In the latter case it is of interest to compare the curves obtained by Seeger and Trauble[18] for zinc (see Fig. 14). Curve P-1-3 appears similar to the room temperature curve for zinc; curve P-5-1 appears similar to the low temperature curve for zinc. The impurities in beryllium apparently affect the work hardening characteristics of beryllium in a manner similar to the effect of temperature for zinc.

3.2.6 Comparison of Results to Other Workers

Considerations of the influence of the method of testing suggested that the limited ductility observed by Tuer and Kaufmann[2] might have arisen from their technique. To verify this a single crystal (V-C)* similar in purity to the crystals used in their investigation was fashioned into a tensile specimen. The machining and testing techniques used were identical to P-1-3. The ductility value obtained was the same as that found by Tuer and Kaufmann (see Table III). With regard to these relatively impure crystals and in comparison to our method of testing, it was concluded that no particularly adverse conditions existed in the test methods of Tuer and Kaufmann. Thus, since the impurity level of V-C and the crystals used by Tuer and Kaufmann was appreciably higher than the zone melted ones, the limited basal slip observed was caused by the relatively high concentration of impurities.

In the work of Garber, Gindin, and Shubin[4] all the crystals (with one exception) deforming by basal glide failed at strains less than 2% (overall elongation). A crystal oriented at $\chi_o = 20°$ failed at 11% overall elongation. The vacuum distilled beryllium used in this work is apparently purer than that of Tuer and Kaufmann and no doubt is comparable to some of the crystals used in the present investigation. One

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* This crystal kindly supplied by Dr. S. Gelles of N.M.I.

would have expected, in view of the results reported herein and previously,[6] that there would have appeared greater ductility values than were observed by Garber, Gindin, and Shubin. Since the experimentally observed ductility values presently obtained can never surpass the ability of the material to plastically deform, the relatively poor results obtained by them must reflect singly or in combination their method of crystal preparation and testing.

Possibly contributing to the low values obtained was the practice of lightly polishing with grinding paper the crystal surfaces prior to testing. Also the gage sections tested were rectangular with the basal plane arranged so that the plane rotation accompanying deformation placed the narrow side of the rectangular cross-section in bending. Since bending is conducive to fracture this is a particularly adverse arrangement. That is, the effect of bending would have been minimized had they arranged the basal plane orientation such that the wide surfaces of the crystal were the ones subjected to bending. It is of particular interest to note that the crystal, at $\chi_o = 20°$, showing the 11% elongation strain was cut from a crystal thinner than the rest and as such had a square rather than a rectangular cross-section.

3.2.7. Impurity Level for Basal Plane Ductility

A rough estimate was made of the probable impurity content of P-1-3. The total impurity level was not considered to be particularly low especially when compared to the analytical results reported on vacuum distilled beryllium[19]. Thus, it appeared that exceptionally low total concentrations of impurities were not required to obtain extensive basal glide. For this reason commercial purity grade Pechiney flake, vacuum cast and extruded, was converted into a single crystal and its ductility evaluated.

The single crystal, designated P-5-1, was prepared in one pass by seeding the polycrystalline rod to a crystal whose basal plane was oriented at 45° to the rod axis and moving the molten zone at about 1-1/2"/hour. This speed of zone refining was three times the normal rate and is expected to minimize impurity removal. The crystal was spark discharge machined and electro-chemically polished into a tensile specimen and tested in a manner similar to P-1-3. The results of tensile testing are shown in Table III and Figure 10.

Since this result gave further evidence that extensive basal plane ductility is possible even in commercial purity Pechiney beryllium, there arose the question of the effectiveness of subsequent zone refining passes.

One can examine, for the purpose of determining the effectiveness of multiple pass zone refining, the ductility results of the single pass crystal, P-5-1, (82% overall elongation) and the five pass crystal, P-1-3, (140% overall elongation). In comparing these two crystals the beneficial effect of multiple passes appears evident. Not only did P-1-3 extend considerably more up to the termination of the test, but even then did not fail in the gage section, indicating that its ultimate potential for basal glide was not realized. Also, on the basis of the fracture criterion analysis, (sec. 3.2.5, crystal P-1-3 had an experimental fracture constant of 1.9×10^{15} dynes^2/cm^3 while the value for P-5-1 was 5.9×10^{15} dynes^2/cm^3) appreciably greater ductility was to be expected from P-1-3 than was obtained.

It is only in comparing crystals P-5-1 and P-1-3 (both showing 82% overall elongation) that one might question the effectiveness of multiple zone passes. It has been pointed out previously, however, that there were several factors attributing to premature failure of crystal P-1-3. A direct comparison, therefore, is not necessarily valid. In fact, the comparison between these two crystals exemplifies the observation that since so many factors other than purity influence the extent of

ductility in tests such as these, speculation on the meaning of absolute values of ductility in terms of purity must be done with discretion.

One aspect, however, of the effectiveness of purification is very clear and that is the appearance of an easy glide region in crystals of greater purity (additional zone passes). In a previous section (3.2.5) it has been described how important this particular effect is in contributing to ductility in beryllium and as such must be considered as a primary advantage of continued purification.

3.2.8 Relation of Single Crystal Ductility to Polycrystalline Ductility

The appearance of extensive basal flow in a crystal (P-5-1) with one zone pass and nominally of commercial grade Pechiney flake beryllium suggests that the principal cause for the limited ductility observed in polycrystalline beryllium of this purity is the constraining effect of the grains themselves. The constraints arise out of the variation of orientation from grain to grain and the limited slip systems available in beryllium for plastic deformation. This produces non-homogeneous deformation within individual grains, a condition which, for grains oriented for basal flow, leads to the formation and splitting of bend planes and hence the formation of cracks.

Based upon the results of single crystal deformation studies it is not possible at present to predict what ductility in polycrystalline material might arise out of appreciably purer beryllium. It can be pointed out, however, that the shear stress-shear strain curves of beryllium of a purity greater than P-5-1 show that the purer material exhibits a lower yield stress and lesser work hardening. Both of these properties are expected to increase the appearance of ductility in polycrystalline material, first by decreasing within a grain the normal stress and second by decreasing the shear stress increment. It is the magnitude of these terms which determine the extent of ductility that can be realized when bend plane formation and splitting is expected to occur; the lower the value the greater the ductility.

Further, one zone pass has been shown (see section 2.2) to remove precipitates felt to be responsible for limiting ductility. Thus, additional zone refining could be expected only to improve this result.

3.3 Compression

In order to evaluate the effect of zone purification on the critical resolved shear stress for prism plane slip, two single crystal compression tests were performed. Since none of the zone refined crystals produced to date was suitable for evaluating prism plane slip in tension (having been oriented for basal slip), it was necessary to make use of small compression specimens extracted from one of the quarter inch diameter, eight pass bars. By suitable orientation it was possible to attain a specimen with the compression axis parallel to the basal plane and at an angle of 30° to the normals of two prism planes.

It has been previously observed that for beryllium the CRSS in tension is roughly half that observed in compression[1,2]. This has been attributed to restriction of flow by frictional forces on the compression faces. Because of this observation, a similar compression specimen was formed with the compression axis at an angle of 30° to the normal to the basal plane, thus permitting an evaluation of the CRSS for basal slip on a corresponding compression specimen.

In both cases the specimens were rectangular parallelepipeds having cross sections approximately 0.125 inches on a side and 0.180 inches high. These specimens were cut from the quarter inch bars by spark discharge machining, followed by careful mechanical polishing and etching of the faces to remove surface damage resulting from the spark machining.

The specimens were then tested in compression in an Instron tensile machine using accessory compression apparatus. The CRSS for the eight pass beryllium samples were found to be 1100 psi on the basal plane and 6500 psi on the prism plane.

It appears, therefore, from these results that purification has not altered the ratio between the stresses for basal and prism glide. It is also noted that the value observed for basal glide is approximately twice that observed for similar purity material in tension and is thus in rough agreement with the variance observed previously between compression and tension values.

4. POLYCRYSTALLINE STUDIES

4.1 Introduction

In order to evaluate the effects of zone refining on the ductility of polycrystalline beryllium, it was necessary to devise methods for converting single crystals of beryllium into fine grain polycrystalline aggregates. Single crystals invariably were formed in multiple pass zone refined beryllium because of the slow solidification rates used. The usual metallurgical approach for reducing grain size of a cast material is by cold work and recrystallization.

There appears, however, limited information about the deformation and recrystallization of single crystals of beryllium. Tuer and Kaufmann[2] extruded a single crystal and observed no recrystallization. Yans, Wolff, and Kaufmann[20] reported on the rolling of single crystals of beryllium oriented with the basal planes perpendicular to the plane of rolling and with up to 50% reduction in area. This latter work was undertaken as part of a study to understand the derivation of texture in beryllium sheet so that techniques might be developed to produce less preferred oriented sheet. In their reported work no attempt was made to subsequently recrystallize those crystals which were deformed at 600°C. These crystals exhibited no recrystallized grains after rolling. The crystals deformed at 1060°C recrystallized at the hot-working temperatures and yielded a coarse grain size (estimated from a photomicrograph as between 200-800 microns) with a "not highly oriented structure"[20]. In the present work the first consideration was for the production of a small grain size since under this condition both experience and the predictions of the Stroh fracture analysis indicate that ductility is favored.

In the deformation and recrystallization of metals the final grain size, exclusive of the temperature contribution, is determined by the amount of cold work that can be introduced, the greater the amount of cold work the finer the grain size. The amount of cold work, however, is important in relation to the extent of "fragmentation" of the crystals or the amount of recrystallization nuclei developed. The latter appears favored by the activation of as many deformation modes as possible. In starting with polycrystalline material the variable orientations and grain boundary restriants result in the operation of multiple deformation modes during cold-working. In a single crystal of beryllium, however, which is so plastically anisotropic, the activation of relatively large amounts of multiple deformation modes requires that deformation be done at high temperatures. That is at temperatures where prism and basal slip are equally likely and where pyramidal slip can occur. At high temperatures (but below the recrystallization temperature) recovery phenomena are expected to reduce the number of recrystallization nuclei. however, the desire to minimize the possiblity of cracking in the limited amount of refined beryllium available required that only the "higher" temperature deformation be considered.

In order to control the recrystallization process and also minimize cracking it was decided that the deformation should occur at a temperature just below that at which recrystallization during deformation was expected to occur. This would permit recrystallization to be subsequently carried out at simple time-temperature schedules.

The methods available for the cold working were swaging, rolling, extrusion, and forging. Rolling was chosen, principally, because of the ease at which the temperature during deformation could be controlled and the multi-directional ways in which the deformation can be introduced.

The initial crystal orientations were chosen to permit slip on the basal prism, and pyramidal planes. Reduction of areas of 90% were arbitrarily chosen as being a "large" amount of deformation conducive to producing a fine grain size.

The testing procedures for evaluating the ductility of any polycrystalline beryllium that was formed was to be determined by the degree of preferred orientation of the sheet formed by rolling. If a somewhat random texture was developed, simple tensile testing of the sheet would be adequate. If, however, a high degree of preferred orientation of the basal planes in the plane of the sheet was developed, bend testing under conditions of plane strain (width to thickness ratio greater than 8 to 1)[21] would be used.

4.2 Procedures and Results

The first rolling experiment was carried out on a one pass zone refined piece of commercial grade Pechiney beryllium. The crystal was 1/4" in diameter and about 3/8" of an inch high with the basal planes oriented at 45° to the cylindrical axis. The crystal was "canned" in a heavy wall mild steel container and rolled with the cylindrical axis perpendicular to the rolling direction. The temperature of rolling was 700°C. The container was reduced 5-10% per pass to a total reduction of 90%. The direction of the first pass was arranged to coincide with one of the slip directions in the crystal. Upon each successive pass the container was rotated 120° to a new rolling direction. It was reasoned that the operation of new slip systems would enhance the "fragmentation" process.

The steel container, after rolling, was stripped off in nitric acid. The beryllium upon metallographic examination revealed a cold worked structure with no recrystallized grains. The cold worked beryllium was annealed at 700°C for 1 hour and 4 hours and at 800°C for 1 hour. The resulting structure was examined metallographically and by x-ray diffraction. At 700°C for 1 hour there was no evidence of recrystallization;

at 700°C for 4 hours, very slight recrystallization. At 800°C for 1 hour the structure was completely recrystallized (equi-axed grains) to roughly a 50 micron grain size. The orientation of the crystals appeared, by a polarized light analysis, to be highly oriented with the basal planes lying in the plane of the sheet.

Additional rolling studies were undertaken; the materials used are listed:

a) eight zone pass commercial purity Pechiney beryllium 1/4" in diameter.

b) one zone pass NMI vacuum distilled beryllium 1/4" in diameter.

c) four zone pass commercial purity Brush beryllium 1" in diameter.

All three samples were approximately three eights of an inch long and were jacketed in heavy wall mild steel containers with stainless steel covers in such a manner that each sample would be reduced, during the rolling, along the cylindrical axis of the samples. For the eight pass Pechiney material and the four pass Brush material the basal plane was tilted approximately 70° to the rolling plane. For the vacuum distilled material the basal plane was tilted 45° to the rolling plane. The rolling was carried out at 700°C and the procedure used for all three samples was as follows. Initially, reductions of five per cent per pass were made using three rolling directions 120° apart (accomplished by the use of hexagonal jackets), up to a two to one reduction from the starting thickness. The remaining passes, up to a final ten to one total reduction, were then made in a single direction using ten per cent reductions per pass. After rolling, the specimens were removed from the jackets by etching in aqueous nitric acid.

The samples were examined metallographically and by x-ray diffraction techniques both in the as-rolled condition and after annealing at 700°C and 800°C. In the as-rolled condition all three samples showed a heavily deformed structure and exhibited solid x-ray diffraction rings.

After annealing at 800°C for one hour the specimens showed an equiaxed grain structure and correspondingly spotty diffraction rings. The grain size for the four pass Brush material was on the order of a 50 micron average grain diameter; the eight pass Pechiney material was coarser (100-200μ) while the vacuum distilled material was very coarse and non-uniform. Although a complete analysis of the texture of the specimens was not made, these samples were apparently very highly oriented, with the basal planes parallel to the plane of the sheet. Examination with polarized light showed nearly complete extinction of all grains for a full 360° rotation of the specimens.*

In an attempt to reduce the degree of texturing observed in the three initial samples, a fourth specimen of the four pass one inch Brush bar was rolled at 700°C with the basal plane perpendicular to the rolling plane and parallel to the rolling direction. At this orientation it was reasoned that little basal slip would occur. The rolling was all done in one direction with 5% reductions per pass up to a two to one reduction followed by 10% reductions per pass to a final ten to one total reduction. This material was recrystallized for two hours at 750°C to produce a mean grain diameter of approximately 70 microns. Metallographic examination under polarized light showed extinction over much of the sample. The grains which did not show extinction, however, were in somewhat localized banded areas. It appeared that some recrystallization occurred during rolling such that the banded areas represented certain recrystallized grains which were further deformed and then recrystallized to produce localized areas of different orientation.

Crude bend tests were performed on these specimens. The first four pass Brush sample exhibited 24° and 15° bends about a one eighth inch mandrel while the second four pass Brush sample exhibited a 30° bend. These specimens had a ten to one width to thickness ratio. The eight pass Pechiney material having a twelve to one width to thickness ratio underwent a 60° bend, producing about 10% strain at the outer

*View perpendicular to plane of the sheet

surface. Because of the non-uniform cross section and extremely erratic grain size the vacuum distilled material was not tested.

4.3 Discussion

4.3.1 Rolling and Recrystallization

In the present work it has been possible to produce a 50 micron, equiaxed grain size of a highly oriented nature in beryllium by the rolling and recrystallization of single crystals. From the standpoint of a developing high degree of multi-directional ductility (aside from possible impurity effects) appreciably smaller and less highly oriented grains would be desirable. These results, however, must be considered as preliminary since no attempt was made to examine systematically the deformation and recrystallization variables (amount of deformation, initial crystal orientation, temperature and time of recrystallization, etc.) in a effort to obtain both a minimal final grain size and a minimal degree of preferred orientation.

The influence of purity on increasing the final grain size is very pronounced as can be seen when the eight zone pass Pechiney and the one zone pass vacuum distilled beryllium is compared to the crystals developing the 50 micron grain size.* This is no doubt due to the effective lowering of the recrystallization temperature with purity. While it is generally observed for metals that increasing purity makes it more difficult to obtain a uniform fine grain size by deformation and recrystallization, it is hoped that in the case of beryllium higher purity with the attendant increase in single crystal ductility, will permit the development of a fine grain size by allowing larger amounts of deformation to be introduced into the crystals at relatively low temperatures.

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* One pass commercial grade Pechiney beryllium.

4.3.2 Ductility Measurements

The strain (~ 10%) that developed in the outer fibers of the eight zone pass Pechiney beryllium bend specimen prior to fracture occurred under conditions (basal planes highly oriented in sheet and deformation under conditions of plane strain) where zero strain at fracture was to be expected. It is, therefore, felt to be a highly encouraging ductility result but which warrants no further speculation at present.

5. ELECTRON TRANSMISSION MICROSCOPY STUDIES*

The examination of thin beryllium foils with the electron microscope is expected to yield considerable information regarding the behavior of dislocations under stress and ultimately lead to a better understanding of the flow and fracture characteristics of beryllium.

Many of the techniques for thinning metals developed in this laboratory[22] have been used for the preparation of beryllium foils. This involves three stages: first,the initial cutting of the specimen to the desired shape, size, and orientation; second,the jet polishing of a depression in the surface in the desired location; and finally,the electropolishing of the specimen to the point where thin areas are produced at the base of the depression.

In the first stage,spark discharge techniques were used for the slicing and shaping operation. Thin sections ranging from 0.050 to 0.100" were cut from a one inch diameter, four pass zone refined beryllium crystal using cutting speeds of the order of 0.001"/min. Slices approximately .030" thick were then cut from the 0.100" sections producing specimens 0.030 x 0.100 x 3/4". These sections were then electropolished to a thickness of 0.020" and were then ready for the second stage.

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*The work reported in this section was performed by Dr. V. V. Damiano of Franklin Inst. Lab.

Techniques were developed for drilling and punching using spark discharge techniques. Small tensile specimens have been cut from 0.20" thick crystals. The specimens are rectangularly shaped and have the dimensions 0.020" x 0.100" x 0.500". A 0.040" hole is located at each end of the specimen for gripping.

In the second stage of the thinning operation a jet polisher was used. The jet polisher is equipped with two calibrated mechanical stages for manipulating both the specimen and the jet. This allows the accurate placement of a depression at any desired location on the specimen within $\pm$ 0.02 mm. A 20% HNO_3 solution was used as the electrolyte with currents of the order of 0.1 amp. for producing either holes or depressions.

In the third stage the depression is viewed through a microscope while the specimen is electropolished. A solution of 9 parts H_3PO_4, 3 parts H_2SO_4, 3 parts Ethanol, 3 parts glycerol is used as the electrolyte. The polishing operation is interrupted at the first appearance of a hole at the center of the depression. This is accomplished by placing a well collimated beam of light behind the specimen and watching for the first breakthrough of light through the foil. Since the procedure of visually observing the specimen is generally long and tedious, a photoelectric cell was placed in the eyepiece of the microscope to sense the first breakthrough of light. At this time an alarm alerts the operator to remove the specimen from the electrolyte.

Figure 15 shows the structure of a section of the one inch diameter four pass zone refined beryllium. The (0001) plane is approximately parallel to the surface of the film and dislocations and dislocation networks lying in the (0001) plane are seen. For convenience $< 11\bar{2}0 >$ slip directions have been designated AB, BC, and CA. Three-fold nodes typical of hexagonal nets are visible. These are believed to arise from the interaction of two sets of 1/3 $[11\bar{2}0]$ type Burgers vectors. The reaction AB + BC = AC results in the formation of a third dislocation at the point of interaction.

Preliminary experiments have shown that the dislocations do not move under the influence of the electron beam alone as they have been observed to move in other metals. However, when the specimen was heated to 200°C and cycled over a narrow range of temperatures, slow movement of the dislocations was observed.

6. SUMMARY

The significant feature of the present investigation is the demonstration that basal plane ductility in beryllium, is not necessarily limited to the small values that others have observed. Further, the extent to which the basal plane can glide, within the restriction of the type of tensile test used, is related to the amount of impurities present in the beryllium.

It is reasoned that the limited slip observed when basal flow occurs in polycrystalline material is not associated, as previously thought, with the inability of the basal planes to sustain any flow without cracking. Rather, the limited ductility is the result of the grains themselves constraining one another during deformation. This produces non-homogeneous deformation of the basal planes which provides the necessary sequence of events for basal glide to lead to crack formation and propagation at low strains. This mechanism of fracture is expected to operate independent of the level of purity of beryllium. The decrease in yield strength and work hardening with purification, however, would make the operation of the mechanism more difficult and result in greater ductility. The extent to which the increase in ductility can be realized in polycrystalline material cannot be theoretically predicted. This must be determined by testing polycrystalline beryllium prepared from highly refined material.

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7. APPENDIX I

7.1 One Inch Diameter Zone Melting Apparatus

The apparatus for one inch diameter bars, as shown in Figure 16, is essentially a scaled up version of that used for the small diameter bars (see Fig. 1). The bar assembly consists of three titanium chucks, a beryllium gettering bar, the beryllium bar to be zone refined and a magnetically supported carriage. As in the smaller system this assembly is suspended vertically from a hook at the top of the quartz enclosure tube. When a molten zone is established in the beryllium bar the bottom end of the assembly is supported by the magnetic carriage. A pyrex water jacket encloses the quartz tube. The induction coil, a simple four turn helix, is external to this jacket and is powered by a 20 KW 9600 C/S motor generator.

The only innovation in this system with respect to the smaller diameter apparatus was in the use of a rotating fan circulating device rather than a piston pumping device. A sketch of this fan mechanism is shown in Figure 17. A rotating magnetic field, created by means of a mechanically driven split-ring commutator, is established in three solenoids, 120° apart, external to the glass enclosure. This field drives the fan by coupling to a two pole soft iron armature inside the glass enclosure.

A problem to be overcome in this device was the design of a bearing which could be run without ordinary lubrication for long periods of time; the danger of contaminating the gettered atmosphere by a lubricant necessitated this approach. The solution to the bearing problem was found in the use of a glass hypodermic syringe. Since a hypodermic syringe is very closely ground, the inner and outer sleeves perform as an air bearing and as such run for long periods of time without any tendency towards seizure.

7.2 Zone Melting Procedure

The actual procedure used for the zone melting of beryllium, for both one quarter inch and one inch diameter bars, was as follows. The bar assembly (chucks, bars, and magnetic carriage) was assembled and correctly aligned in the quartz enclosure tube by the use of adjusting set screws in each chuck. The whole system was then joined to an all glass vacuum system consisting of a mechanical forepump and a three stage oil diffusion pump. Liquid nitrogen traps were used to prevent back-streaming into the system. The apparatus was evacuated to about 10^{-6} mm of Hg, filled with argon to slightly less than one atmosphere, and hermetically sealed. The apparatus was then placed in the zone melting transport mechanism and the induction coil connected to the power supply. The argon atmosphere was gettered internally by circulating it first over the central titanium chuck, heated to about 900°C, and then over a molten zone in the beryllium gettering bar.

The zone melting was carried out by establishing a molten zone, approximately 1/4" long in the 1/4" diameter bars and 5/8" long in the one inch diameter bars, and traversing this zone along the length of the bar at a rate of 1/2" per hour. The direction of travel was from top to bottom to take advantage of gravity segregation, since almost all impurities in beryllium, including BeO, are more dense than beryllium itself. The zone length was constantly monitored by an operator.

At the completion of the pass, the vapor deposit on the internal surface of the quartz enclosure tube was quite opaque. This necessitated the removal of this deposit before a subsequent pass could be made. The bar assembly, therefore, was removed from the apparatus after each pass, the vapor deposit washed with a 2-5% H_2SO_4 solution from the glass tube, and the whole process repeated for the next pass. Between passes, the beryllium bars were given a bright etch in a aqueous nitric-hydrofluoric acid to remove any insoluble phases agglomerated at the surface of the bar.

At no time was there seen any evidence of reaction between the beryllium condensate and the quartz tube. The deposit readily washed off the inner surface of the tube, and the quartz itself remained perfectly clear. In previous experiences with the zone melting of titanium and zirconium, it was observed that the condensate had to be etched from the surfaces of quartz tube enclosures which were not water cooled. Because of the incorporation of the water cooling jacket, the gettering procedure used, and the negligible leak rate of an all glass system, the danger of any gaseous contamination during zone melting has been reduced to a minimum.

8. APPENDIX II

8.1 SPARK DISCHARGE MACHINING DEVICE

A principal phase of apparatus development in the present contract year was the development of a spark discharge machining device used for preparation of test specimens from the zone refined beryllium.

It was observed that the electrochemical machining technique[23] tried earlier had a strong tendency to perpetuate any dimensional non-uniformities found in the starting material. That is, variations in diameter or axial curvatures in the unmachined bars tended to reflect themselves in the final machined specimen. This difficulty in obtaining precise control of specimen geometry during electrochemical machining appears to be a function of the metal being machined. Non-uniform sections of alpha-brass or zinc can be machined fairly readily to desired sizes and shapes using appropriate etches etc. Thus, the possiblity existed that by selecting suitable electrolytes and electrode shapes the satisfactory electrochemical machining of beryllium could have been perfected. Rather than undertake the sometimes unrewarding brewing of etches and electrolytes, it was decided to develop a spark discharge machining technique to impress the desired shape accurately on the specimens.

While spark discharge machining does result in some surface damage, which must subsequently be removed by a minimal amount of electrochemical machining, the fact that the eroding device need never contact the specimen eliminates the development of gross bending strains, as are likely to be produced if grinding was used as a technique for producing specimens.

A schematic drawing of the mechanical apparatus of this machining device is shown in Figure 18. It is essentially a small lathe adapted in a manner to permit the turning of specimens submerged in a bath of oil. The cutting tool, a brass wheel whose surface has been machined to

the desired profile, is also rotated while submerged in the oil bath. The specimen is rotated at 60 rpm and the cutting wheel at 70 rpm so that the specimen and cutting wheel are slightly out of phase. For convenience the specimen chuck and the specimen can be rotated up out of the oil bath to permit examination of the specimen at any time during the machining procedure.

The electronic circuitry for the spark discharge of this apparatus is shown in Figure 19. It is essentially a variable frequency relaxation oscillator with a filtered D. C. power supply. The current (which determines cutting rate) across the spark gap is controlled by the choice of capacitance and resistance in the oscillator circuit. For high cutting rates and coarse finish, low values of resistance and high values of capacitance are used; for low cutting rates and fine finish, high values of resistance and low values of capacitance are used. The cross-feed on the lathe is controlled automatically by monitoring the voltage across the spark gap between the specimen and the cutting tool. The circuitry for this control is shown in Figure 20 and the operation is as follows. The voltage across the gap increases as erosion takes place. When the control point set by the potentiometric controller is exceeded, the drive motor is energized. As the cross-feed drives the tool toward the work, the gap voltage decreases until it drops below the control point. The controller then deenergizes the drive motor and erosion continus. The single point controller has been modified to widen the control range so that normal voltage fluctuations do not result in exceessive relay chatter.

8.2 Preparation of Specimens

Most of the material to be machined was in the form of 1/4" diameter rods of zone melted beryllium. Several square section rods cut from vacuum cast ingots or from 1" diameter zone melted bars were also machined. The technique described below applies in all cases.

A rod was chucked up in a six screw universal chuck. The screws were adjusted such that the section to be machined was running as true as possible.

The rod was brought up to the rotating form tool which was shaped to cut the desired specimen form (see Fig. 18) by the cross-feed screw. As the work approached the tool the feed screw was rotated by hand very slowly so that the final engagement of the tool and work was by a spark discharge over the small working gap (~ .001") rather than by any direct physical contact. Since the rods to be machined were not perfectly straight nor of uniform cross section, there was usually only one contact point at the beginning of the machining operation. This area of contact was enlarged by the erosion action of the spark discharge. During this time the feed screw was rotated manually so that the average gap voltage during the erosion period was not permitted to go below 100 volts. That is, a limiting clearance between work and tool was maintained by arbitrarily choosing a minimum operating voltage. During this initial machining step the capacitance of the discharge gap power supply was set between 1 - 2 mfd. and the current adjusted by the rheostats not to exceed 4 amps.

Eventually a contact area completely around the circumference of the rod was developed. At this point the operating voltage of the gap became fairly constant since the gap clearance was uniform during a revolution of the specimen. Although the current (hence cutting rate) was variable and determined by the instantaneous contact area between work and tool, the cross-feeding operation could now be automated by using the voltage for monitoring.

To do this the gap voltage, decreased by a voltage divider, was applied to a potentiometric controller whose control point corresponded to some arbitrary gap voltage - usually between 100 - 120 volts on the low side and about 140 volts on the high side. As the specimen was eroded the average gap voltage was increased and when the upper voltage

limit was exceeded, the controller turned on a motor driving in the specimen via the cross-feed. As the working gap distance decreased the average voltage across the gap decreased. When the voltage reached the lower limit the controller turned the motor off. By this method the specimen was automatically machined. The total time the cross-feed screw drive was on was recorded by a timer which was set to stop the cutting at a predetermined dimension of the specimen (usually ~ 0.155" diameterin the gage section). This phase of machining was termed rough - ing - the power setting used was that previously mentioned.

The surface of the beryllium bar now appeared "rough" with evidence of twins and cleavage cracks. Further machining at a lower power setting (0.1 mfd. and current not exceeding 1 amp) reduced the extent of surface damage and of course removed additional material. This step was also automatically controlled and stopped when the gage diameter was 0.135".

The surface was further improved by a "fine" machining step consisting as before of a reduced power setting (.006 mfd., current not exceeding 100 ma). This step was also automatically controlled and stopped when the gage diameter was 0.125".

The chuck and specimen was removed and inserted into an electro-chemical machining device patterned after Greetham and Martin[23]. In this step approximately .010" to 0.020" was removed from the diameter of the gage section and a portion of the shoulder. The gage section revealed no cracks by optical examination nor did Laue photograms reveal surface distortion. Most of the shoulder section was "stopped" off by lacquer and prevented from being acid machined. This left the shoulder section, after the lacquer was removed, accurately machined to mate with the tapered jaws of the tensile testing rig. It is noted, however, that the spark discharge machined surface of the shoulders, even though "fine" machined, still showed evidences of cleavage cracks.

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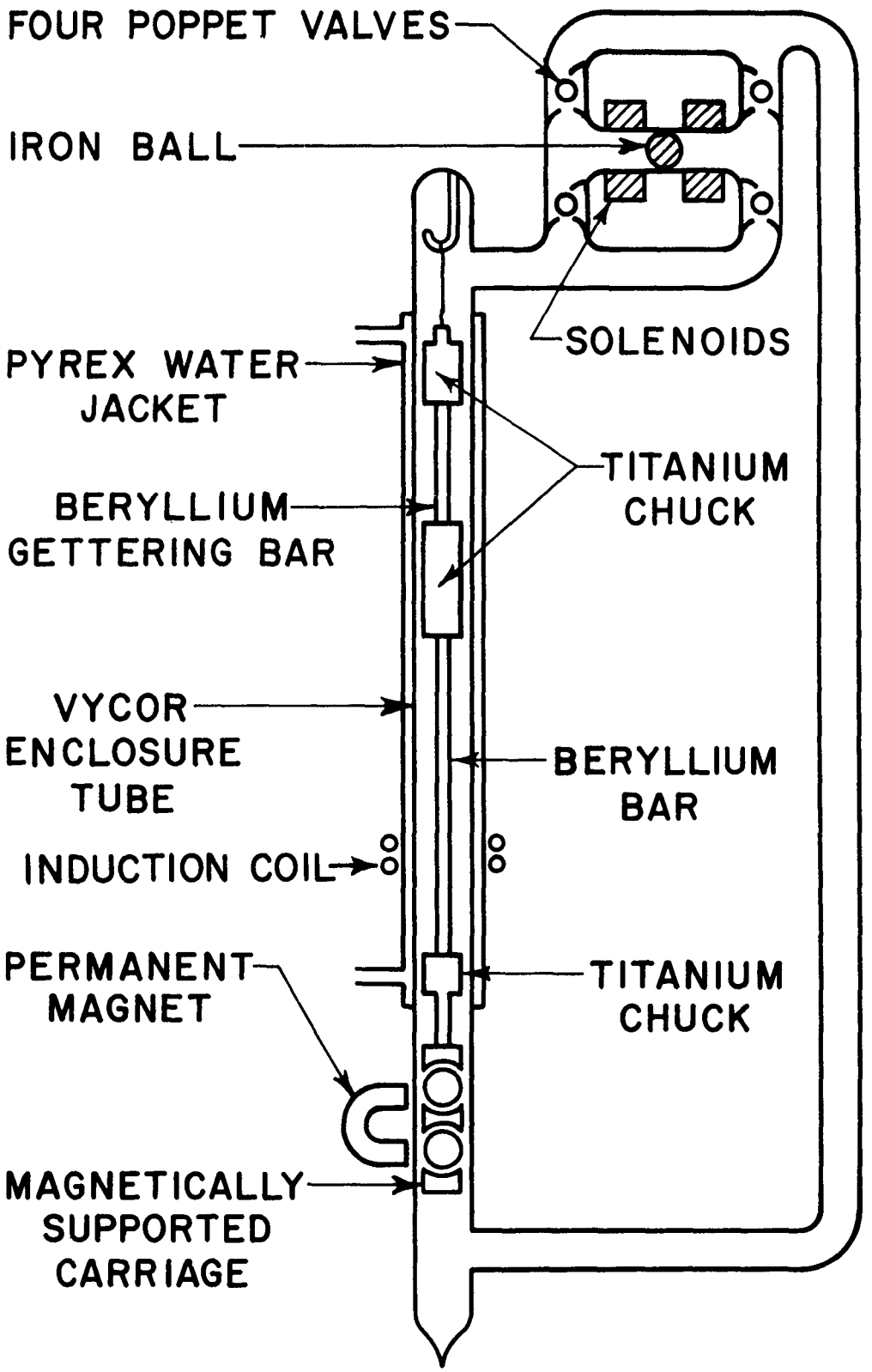

Fig. 1 - Zone Melting Apparatus for One-Quarter Inch Diameter Beryllium Bars

Fig. 2 - One Inch Diameter Beryllium Bar After Two Zone Passes

Fig. 3 - One Inch Diameter Beryllium Bar After Three Zone Passes

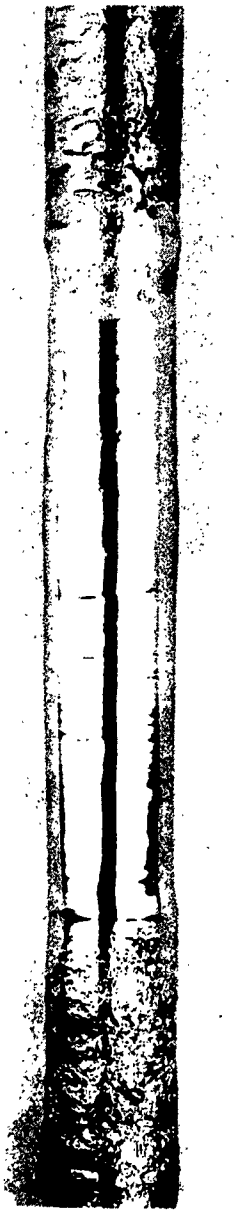

Fig. 4 - One Inch Diameter Beryllium Bar After Four Zone Passes - Bright Etch

Fig. 5 - One Inch Diameter Beryllium Bar After Four Zone Passes - Macroetched to Show Grain Structure

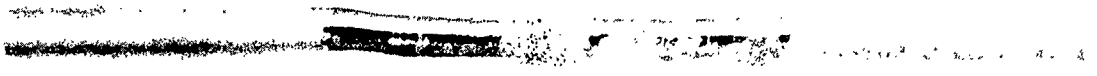

Fig. 6 - One-Quarter Inch Diameter Commercial Purity Beryllium Bar After a Number of Zone Passes

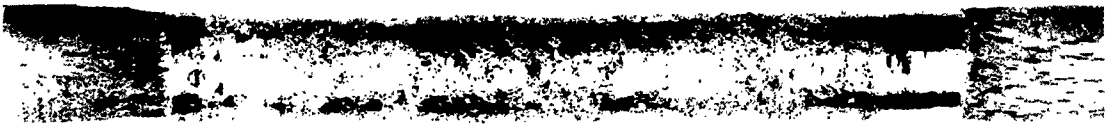

Fig. 7 - One Inch Diameter Pechiney Hot Extruded H.P. Beryllium Bar After Three Zone Passes

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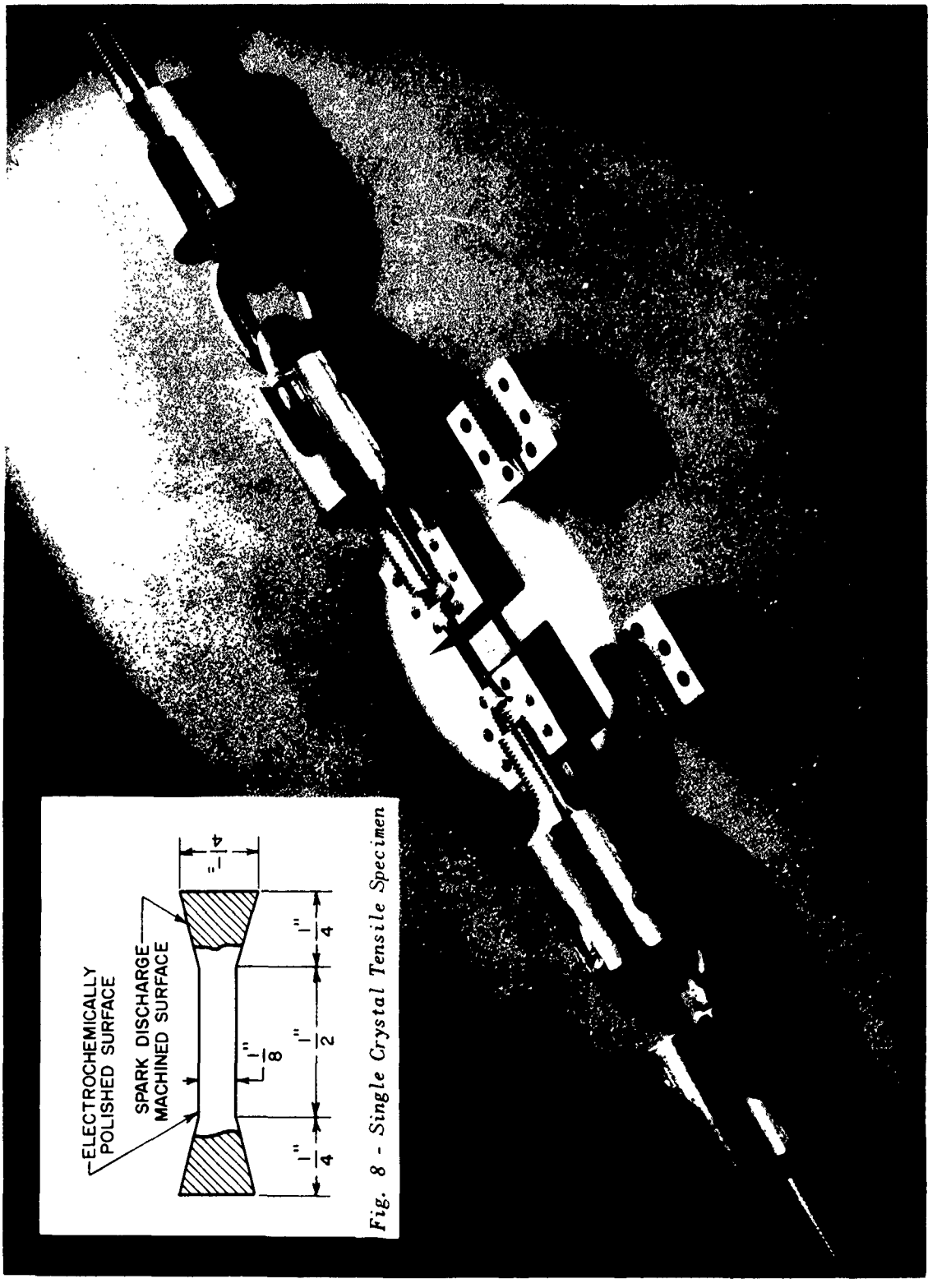

Fig. 8 - Single Crystal Tensile Specimen

Fig. 9 - Single Crystal Tensile Grips and Alignment Chains

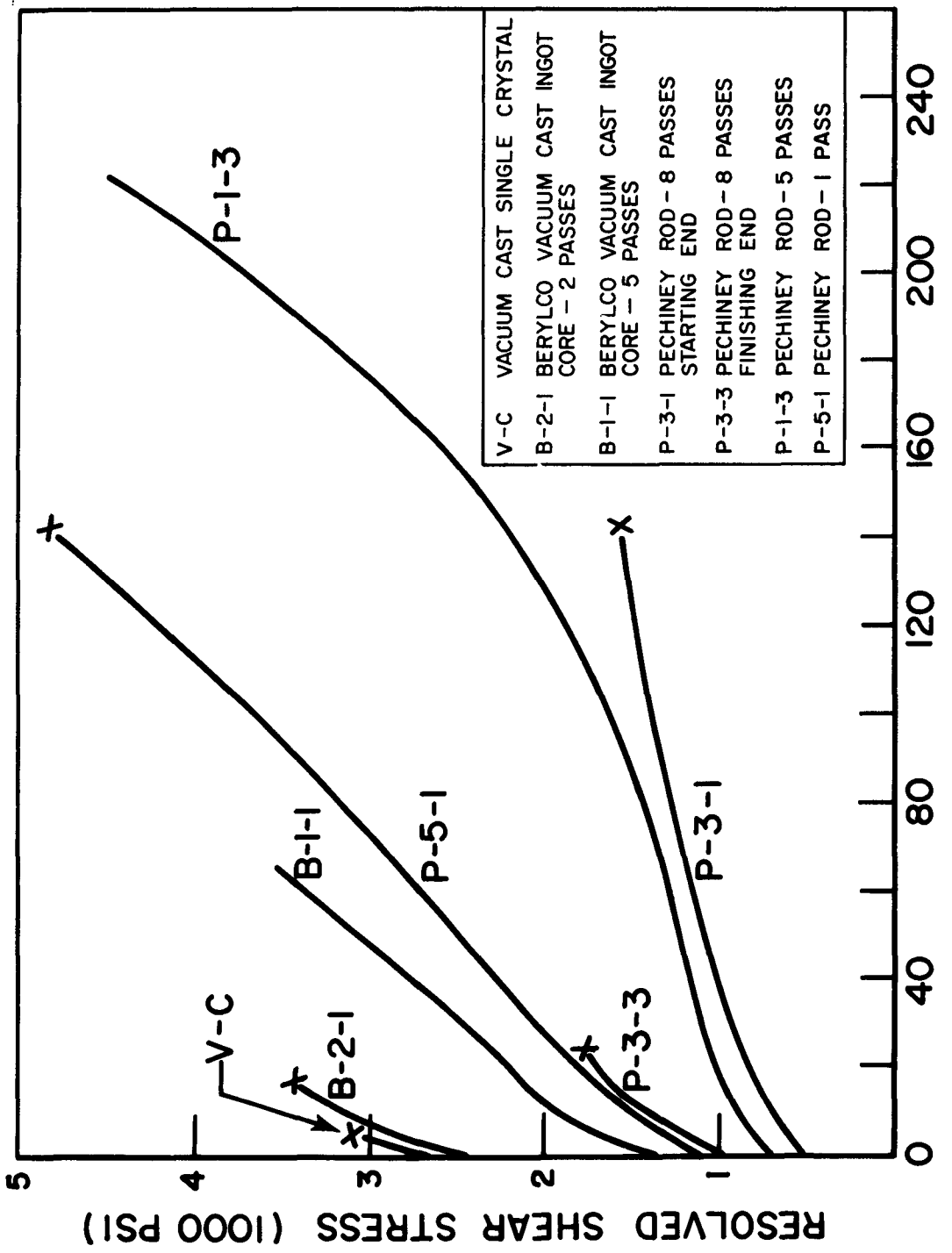

Fig. 10 - Resolved Shear Stress/Glide Strain Tensile Curves for Beryllium Single Crystals. Fracture in Gage Section Denoted by (X).

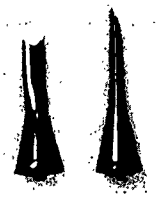

Fig. 11 - Single Crystal Tensile Specimen P-3-1. Portions Rotated 90° to one another.

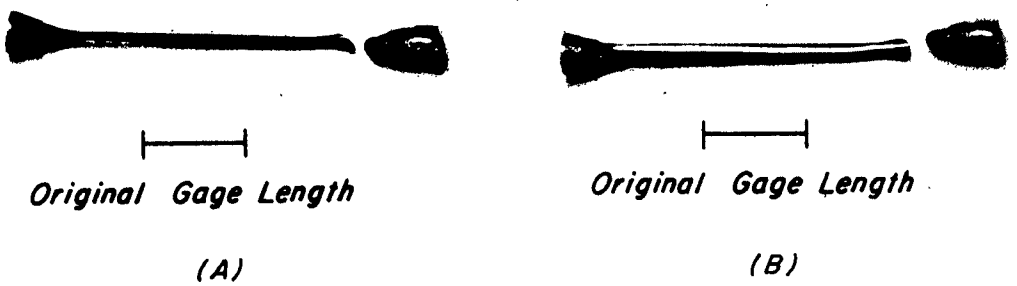

Fig. 12 - Single Crystal Tensile Specimen P-1-3. (a) Viewed along narrow elliptical section. (b) Viewed along wide elliptical section.

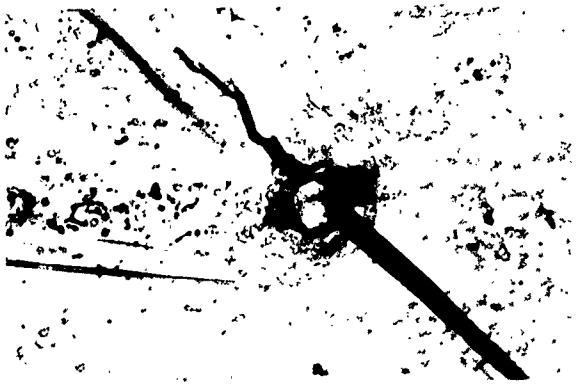

Fig. 13 - Photomicrograph of surface of specimen V-C showing a cleavage crack (major portion along basal plane) associated with an inclusion. Surface as electrochemically machined. X500

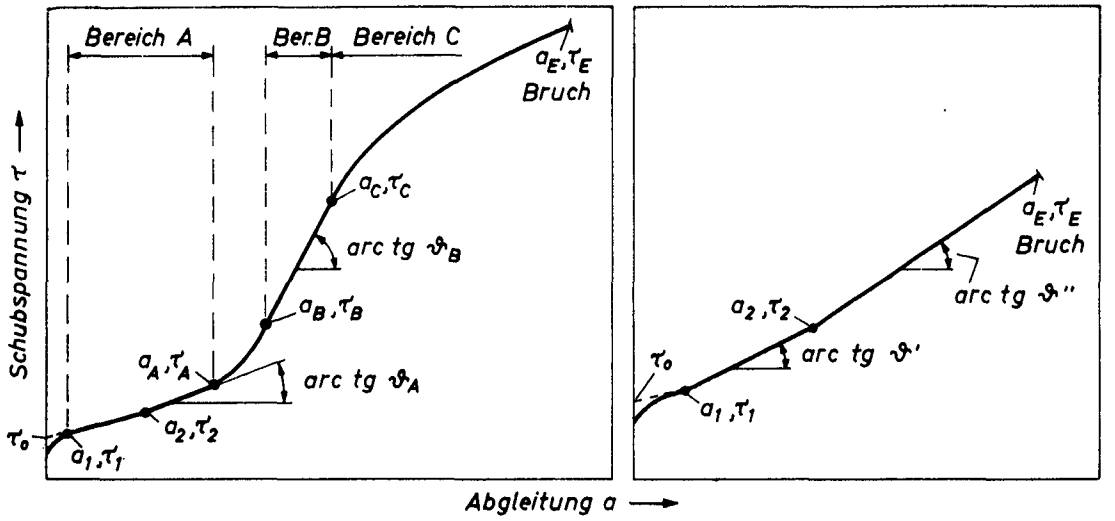

Fig. 14 - Room Temperature, Curve 1 (left) and Low Temperature, Curve 2 (right) Shear Stress-Shear Strain Tensile Curves for Zinc. (Reprinted from Reference 18.)

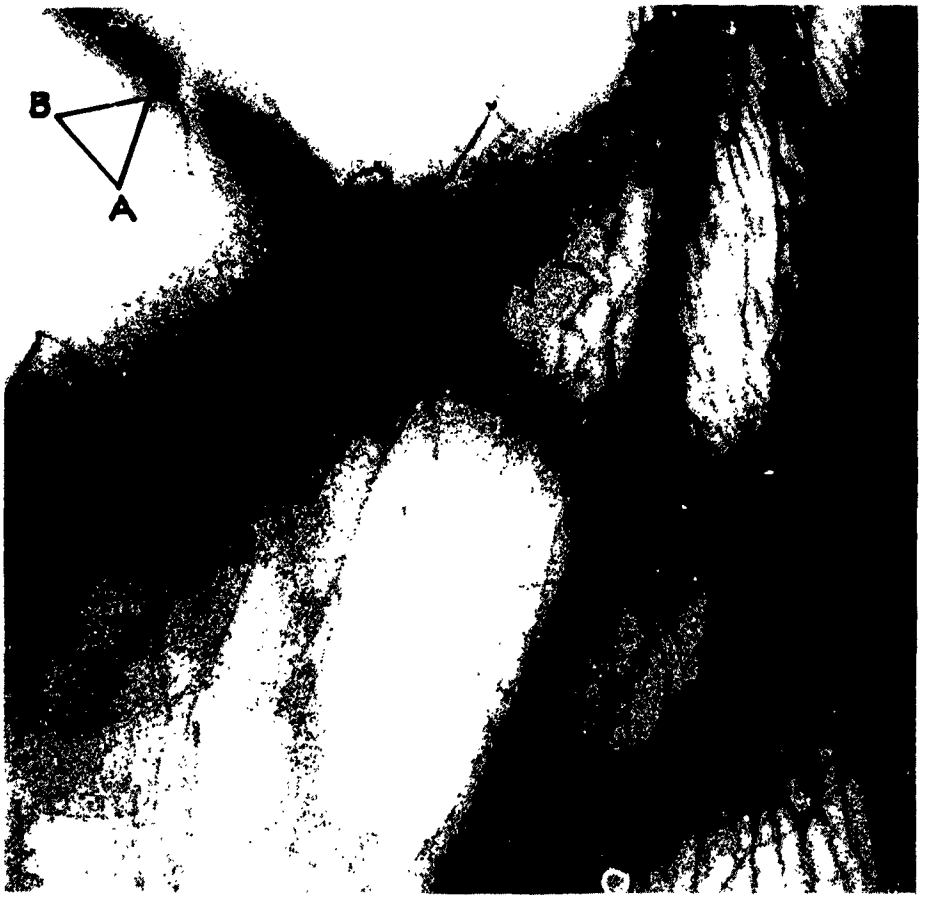

Fig. 15 - Electron Transmission Micrograph of Beryllium Single Crystal, (0001) Approximately Parallel to Plain of View

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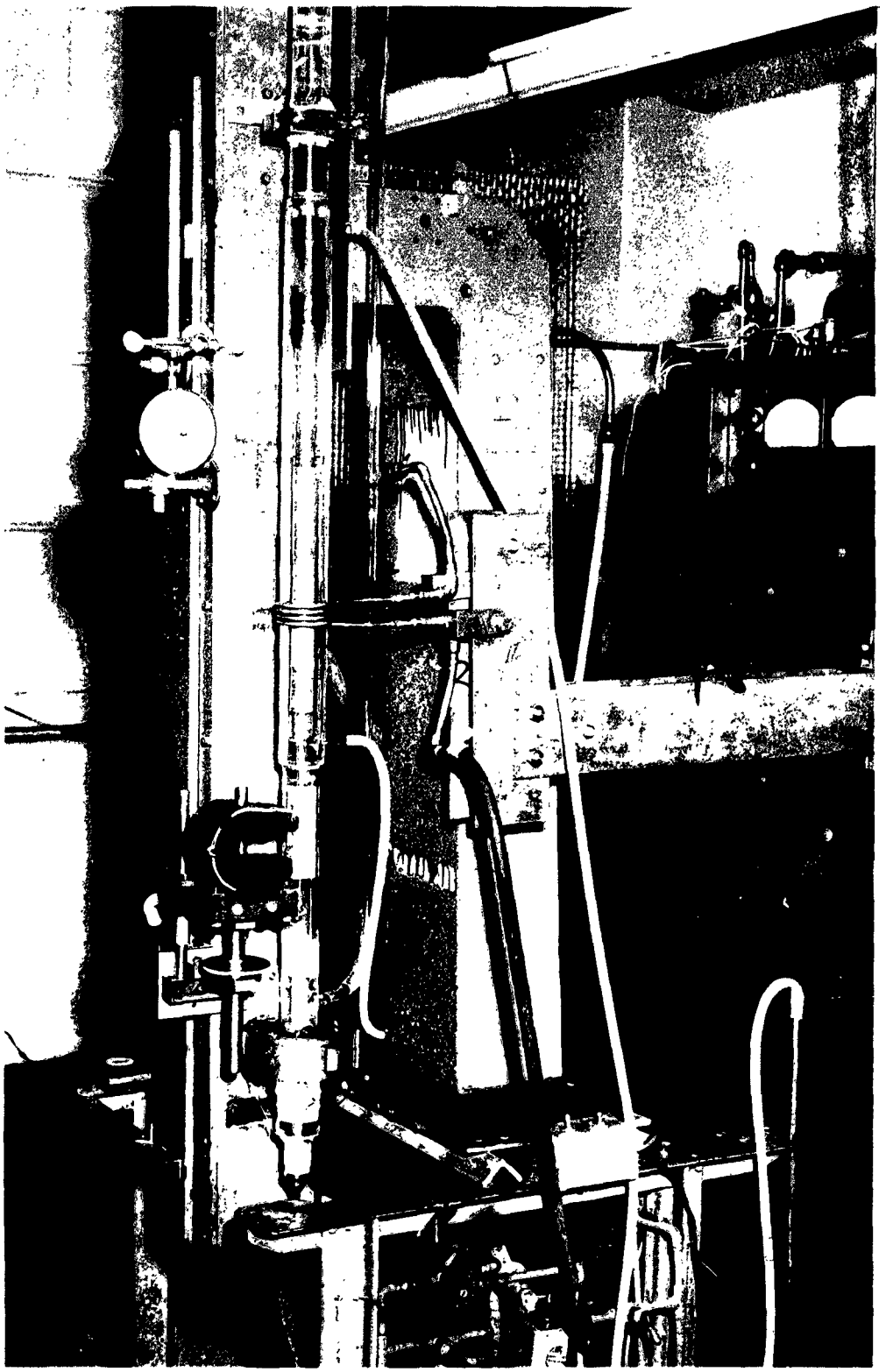

Fig. 16 - Zone Melting Apparatus for One Inch Diameter Beryllium Bars

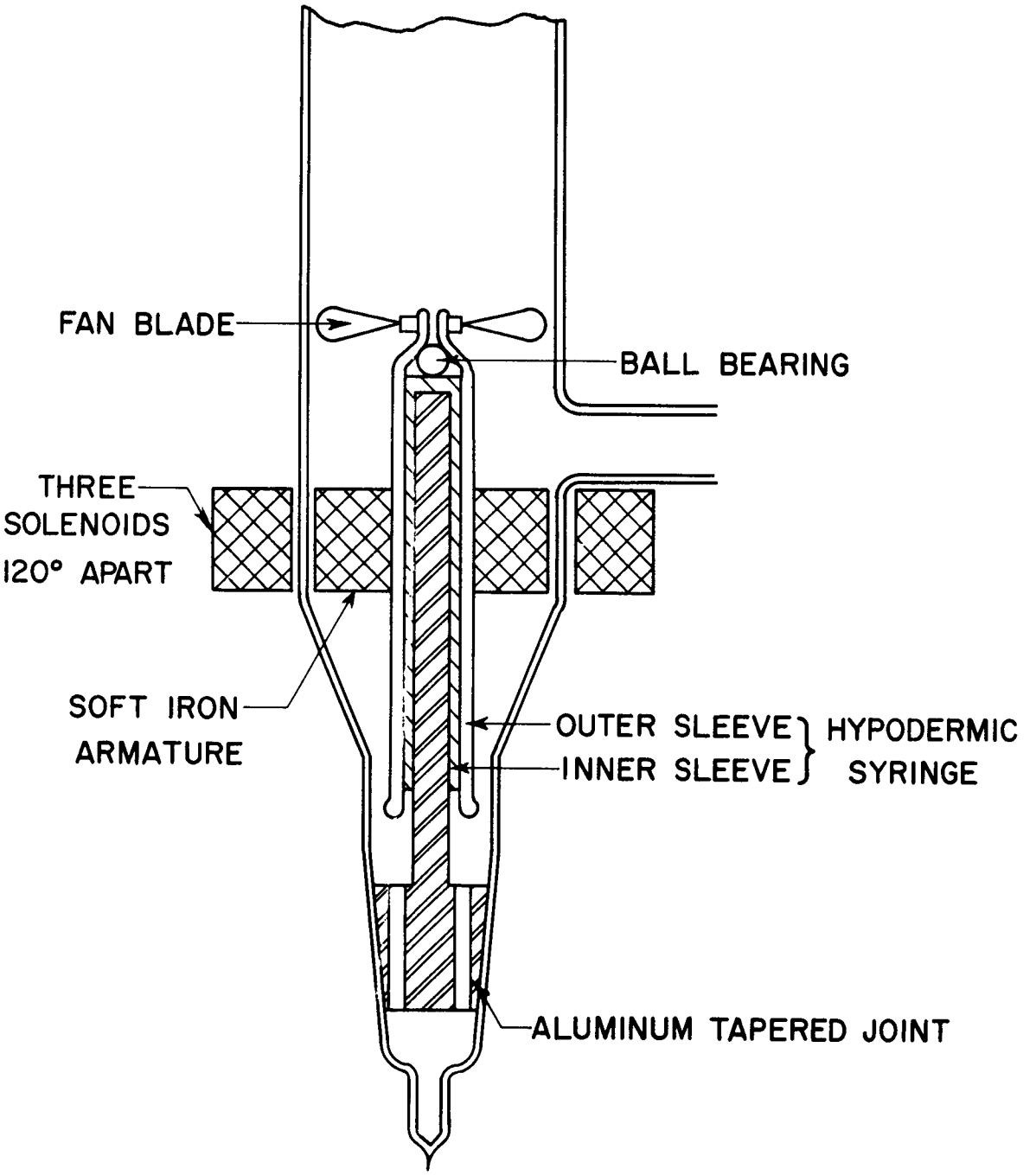

Fig. 17 - Fan Mechanism for One Inch Diameter Zone Melting Apparatus

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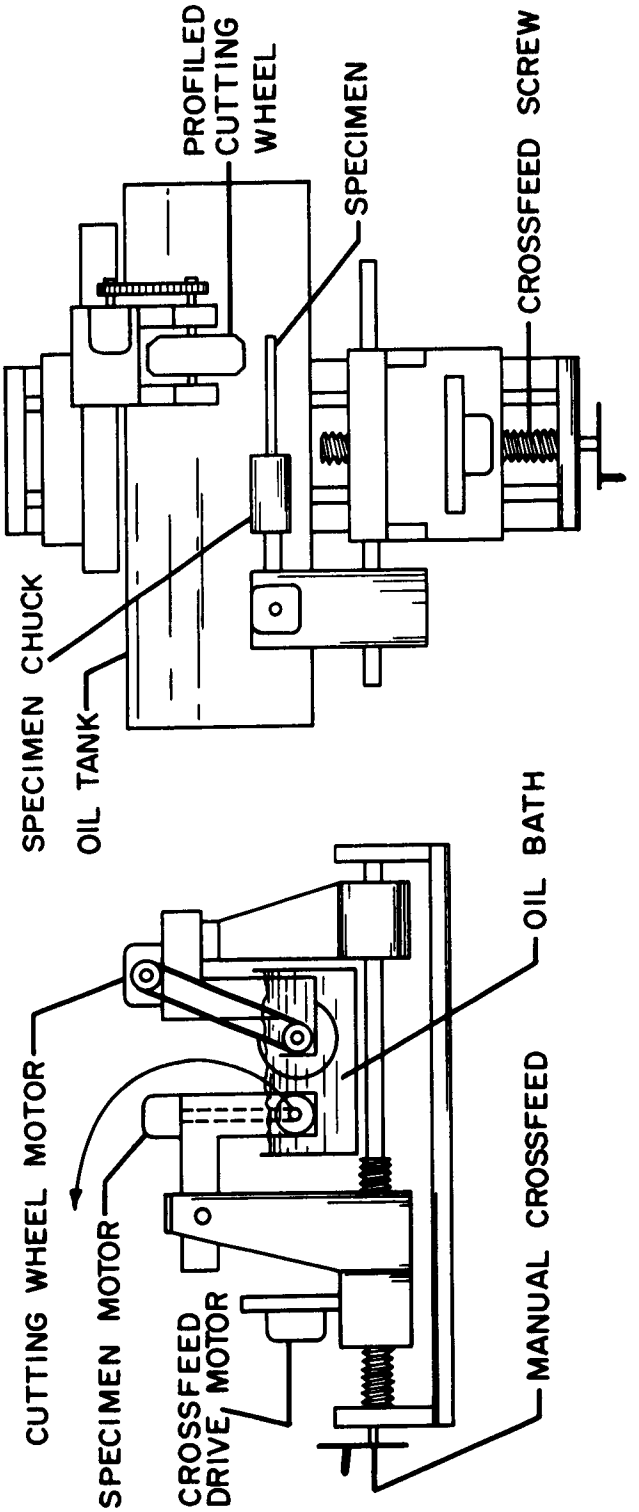

Fig. 18 - Spark Discharge Machining Apparatus

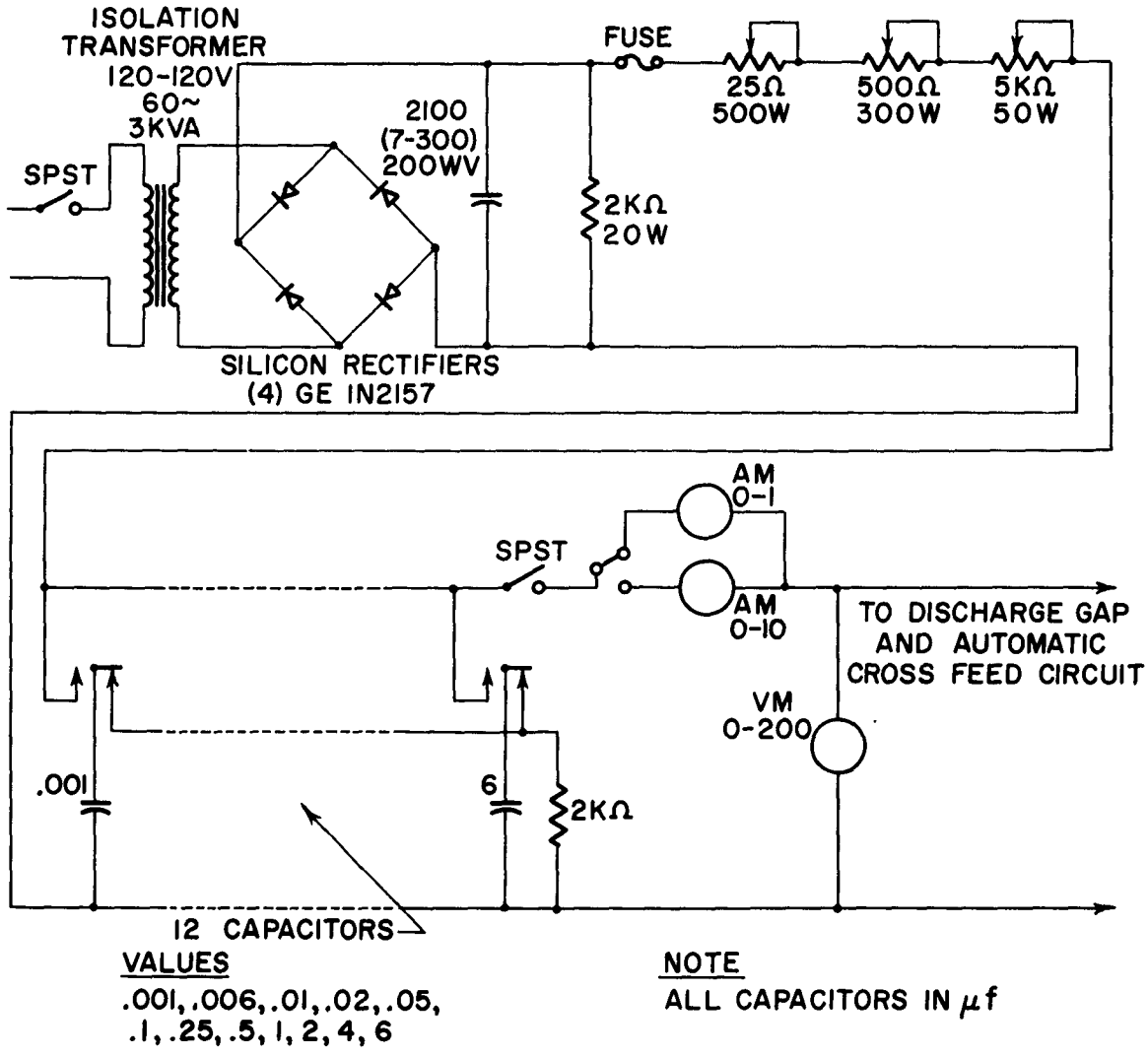

Fig. 19 - Electronic Circuit for Spark Discharge Machining Apparatus

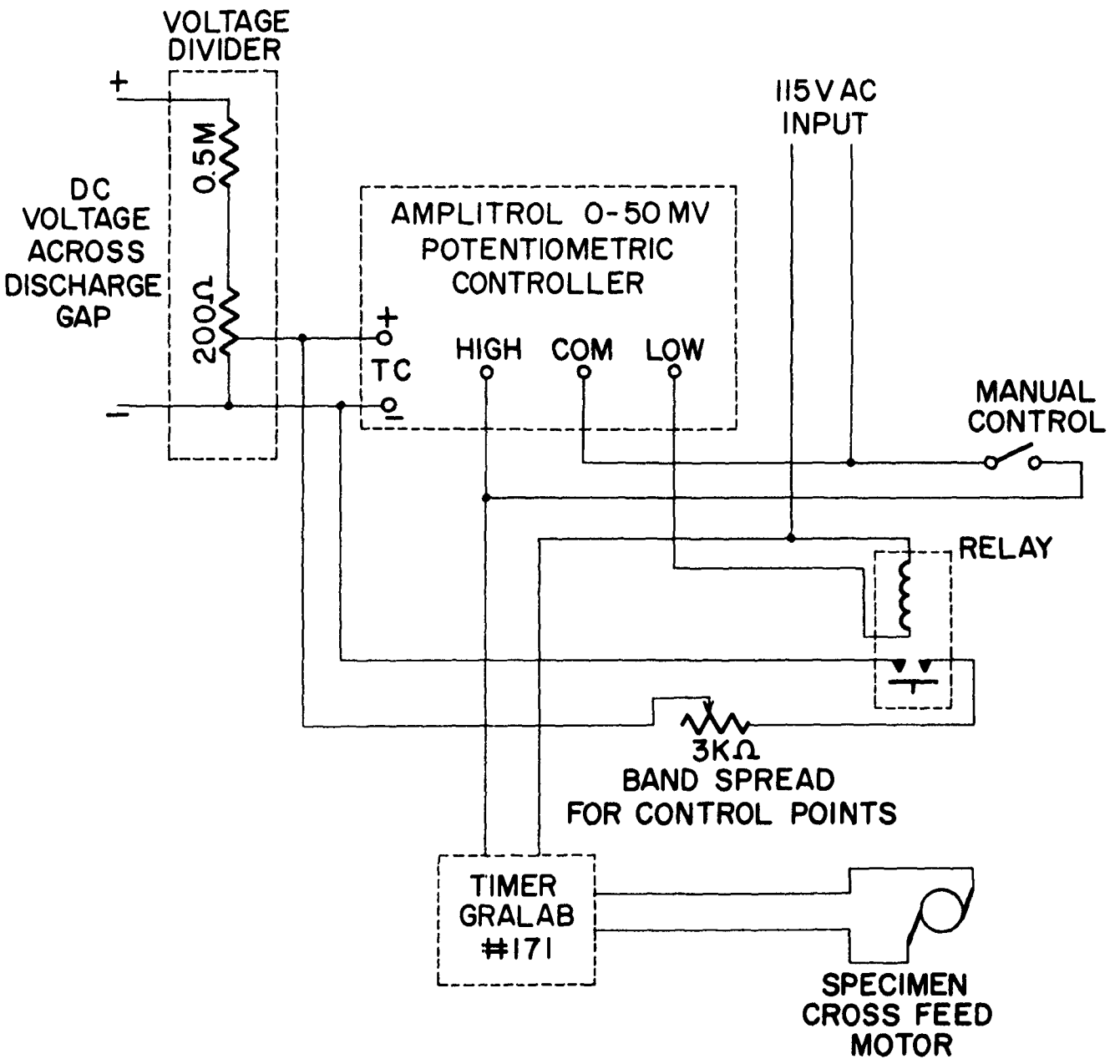

Fig. 20 - Electronic Control Circuit for Spark Discharge Machining Apparatus

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